

# The Journal of the Society of Dyers and Colourists

Volume 77

Number 5



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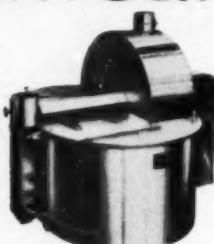
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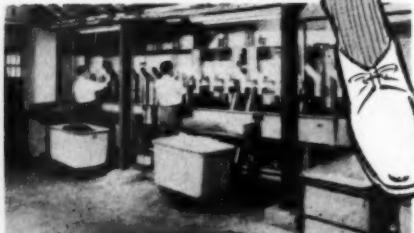


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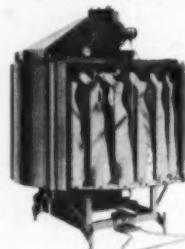
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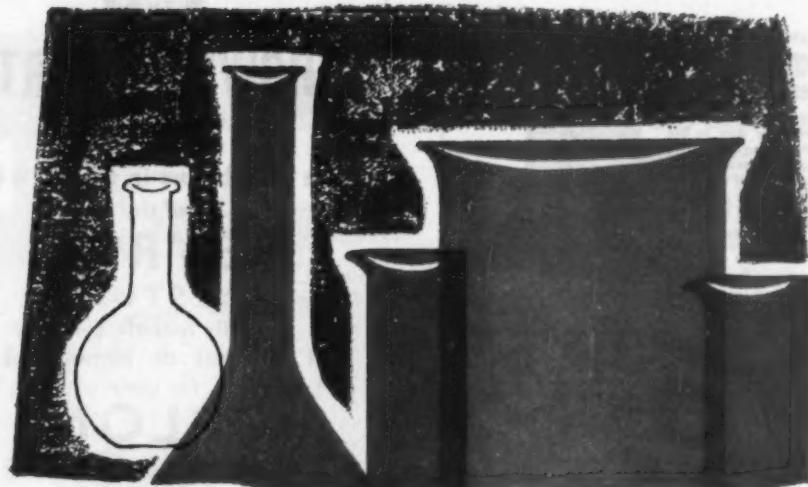


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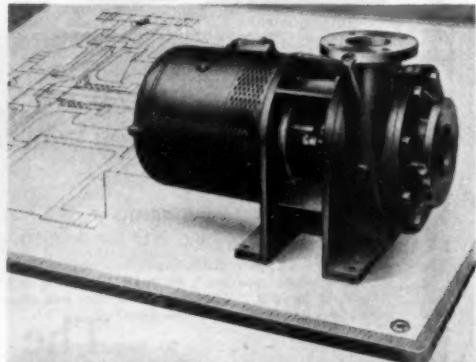
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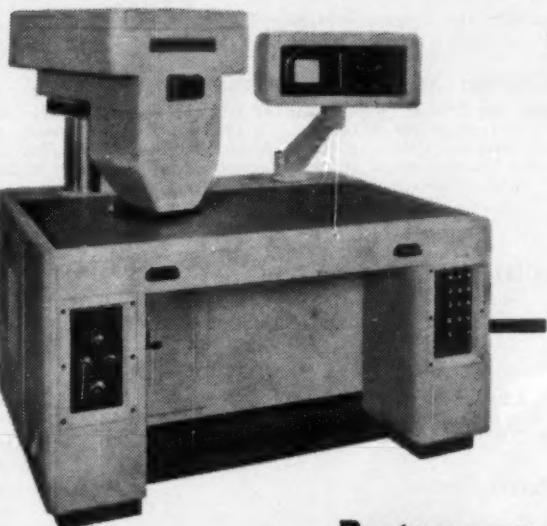
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(Abstracts section only—see January issue, page 2)

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## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURES

- |   |                         |
|---|-------------------------|
| Some Difficulties in the Coloration of Plastics     | <i>J. M. J. Estevez</i> |
| Recent Developments in the Dyeing of Acrylic Fibres | <i>B. Kramrisch</i>     |

### COMMUNICATION

- |  |                                     |
|--|-------------------------------------|
| Effect of Temperature and pH on the Adsorption of Basic Dyes by Wool | <i>C. L. Bird and G. P. Stancey</i> |
|--|-------------------------------------|

*Why not contact*

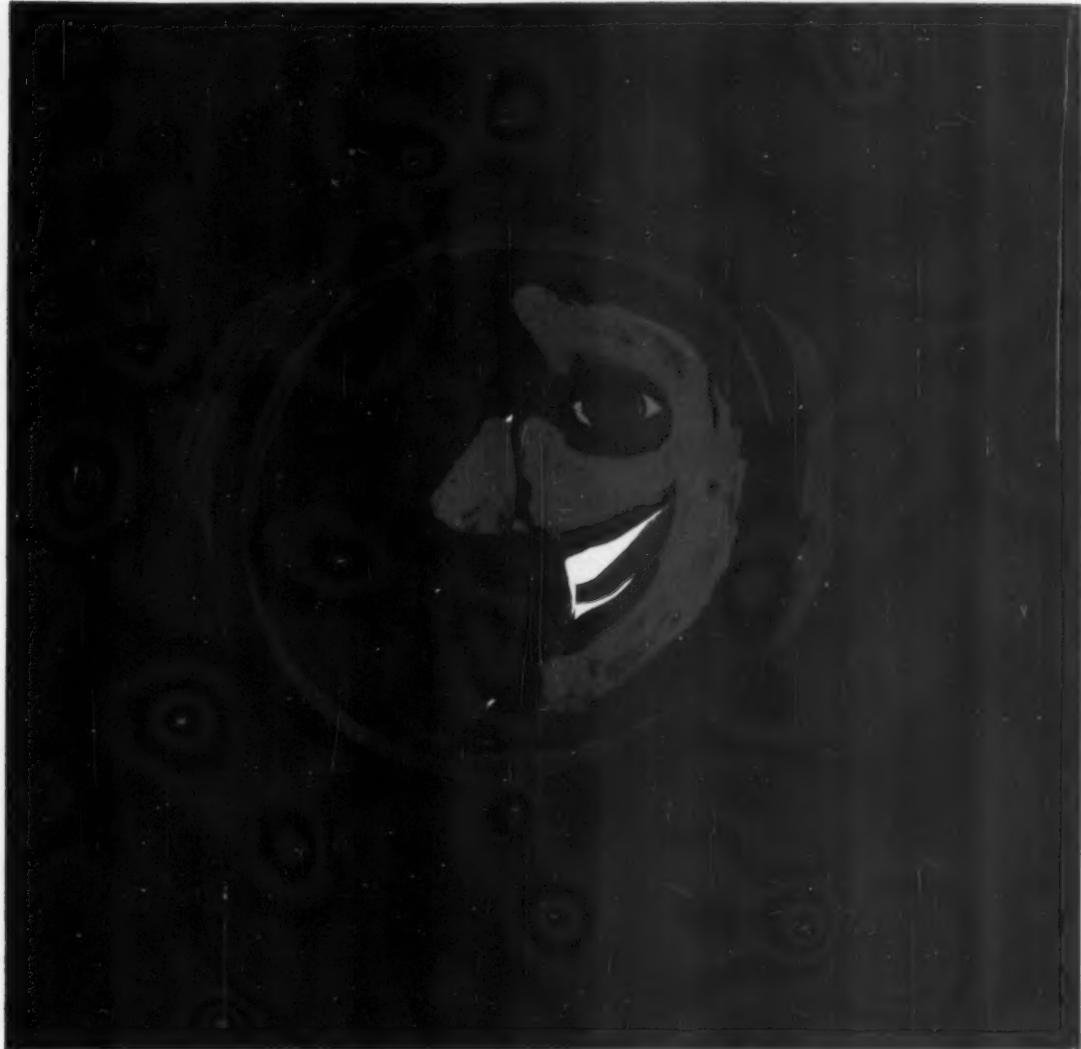
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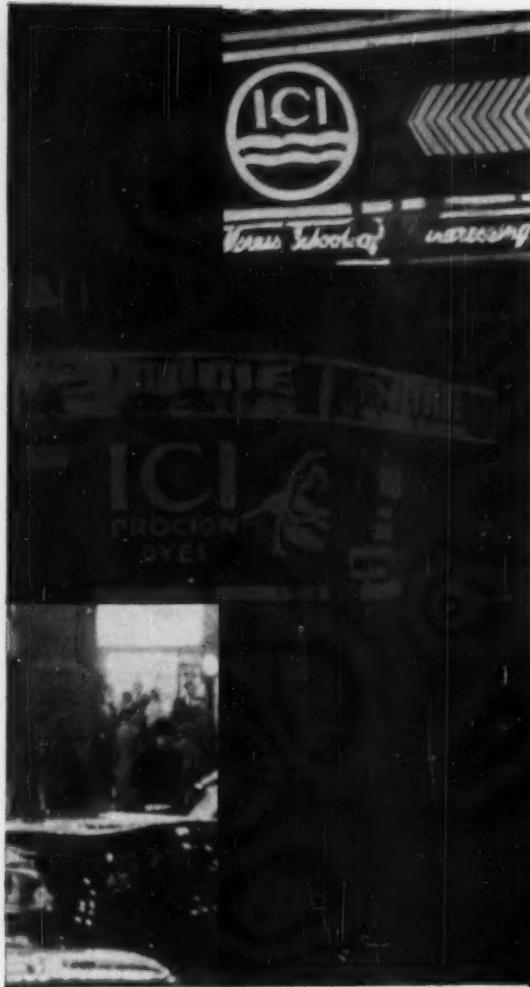
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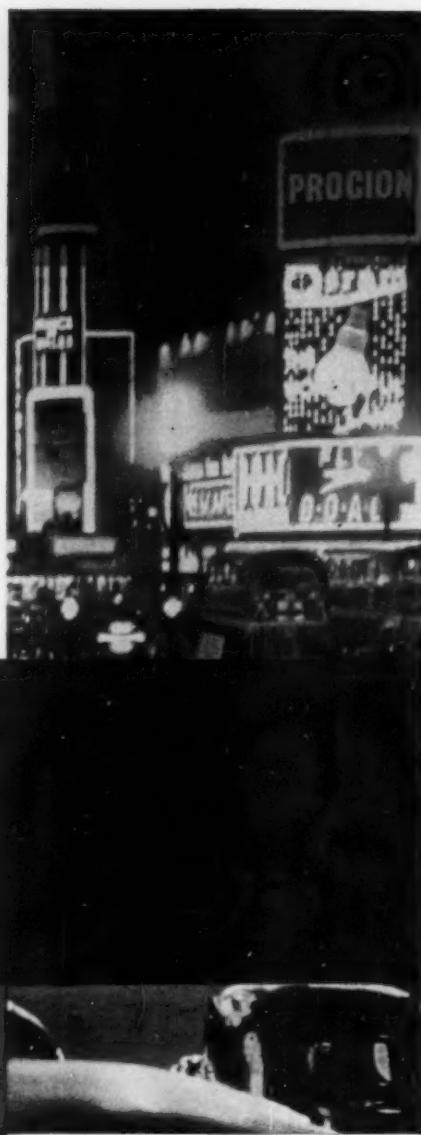
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Volume 77 Number 5

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COMMUNICATIONS

**The Action of Tetrakis(hydroxymethyl)phosphonium Chloride  
on Wool and Hair**

L. S. BAJPAI, C. S. WHEWELL, and J. M. WOODHOUSE

Wool treated with THPC absorbs acid and premetallised dyes more readily than does untreated wool, and addition of THPC to solutions of premetallised dyes increases the rate at which the dye is taken up by the wool. Because of their ability to reduce the S-S linkages in wool, solutions of THPC are effective setting agents, even at pH 1-3. Although oxidised, iodinated, and deaminated wools cannot be set in boiling water, they take a set in THPC solutions. Wool treated with potassium cyanide, however, cannot be set either in water or in THPC solutions.

**Introduction**

Although considerable attention has been given to the reaction of tetrakis(hydroxymethyl)phosphonium chloride (THPC) with cellulose, little information is available about its effect on wool. The present investigation was carried out after a preliminary study<sup>1</sup> had shown that wool treated with THPC had an enhanced affinity for certain acid dyes, and that THPC affected the setting characteristics of wool fibres. This paper provides detailed information on the dyeing and set/supercontraction characteristics of wool and hair in solutions of THPC and related compounds.

**Experimental**

**MATERIALS**

*Wool*

The root ends of Lincoln wool, purified by successive extractions with diethyl ether and alcohol, were used. Dyeing experiments were carried out on commercially scoured light-weight wool flannel.

*Hair*

Chinese human hair was purified in the same way as the Lincoln wool.

*Chemically Modified Wool and Hair*

Small bundles of wool or hair fibres were treated with about 100 ml of various reagents under the following conditions—

*deamination*<sup>2</sup> 43% sodium nitrite in 35% acetic acid; 48 h at 22°C

*iodination*<sup>3</sup> 0.77-N iodine in alcohol; 72 h at 22°C

*oxidation* (a)<sup>4</sup> 1% potassium permanganate in 1% hydrochloric acid; 20 min at room temp.

(b)<sup>5</sup> 1% potassium dichromate in 1% hydrochloric acid; 20 min at room temp.

(c)<sup>6</sup> 2% peracetic acid; 5 min at room temp.

(d)<sup>7</sup> 2-vol. hydrogen peroxide in 2% borax; 18 h at room temp.

(e)<sup>8</sup> 1% potassium iodate in 1% hydrochloric acid; 3 h at room temp.

*caustic soda*<sup>9</sup> (0.11 N); 18 h at room temp.

*potassium cyanide*<sup>10</sup> (0.65%); 30 min at the boiling point.

**REAGENTS**

THPC of commercial quality was purified by crystallisation from alcohol-ether as follows. THPC of commercial quality (100 g) was dissolved in warm absolute alcohol (100 ml) and filtered. Diethyl ether was added to the filtrate until a slight turbidity was observed. The solution was seeded and allowed to stand overnight. The product was collected by filtration, washed with alcohol-ether (2:1) and dried in a vacuum desiccator; the melting point of purified THPC was 147–148°C. Other reagents were of analytical reagent quality except where indicated.

**METHODS**

*Dyeing*

Except where otherwise stated, conventional dyeing techniques were employed.

*Determination of Set/Supercontraction Characteristics*

Fibres of known length were extended in a frame by 40% in water and then boiled in water or a solution of THPC for a given time. The stress on the fibre was then removed and the slack fibre boiled freely in water for one hour. The just-taut length of the fibre in cold water was then measured. The percentage (calculated on the original length of the fibre) of set or supercontraction realised

was plotted against the time of boiling in the extended state. In water, untreated wool fibres supercontract if the time of boiling is short, but take a set if boiled for longer times.

### Results and Discussion

#### DYEING CHARACTERISTICS OF THPC-TREATED WOOL

Samples of wool treated with THPC under various conditions and then rinsed in water were dyed together with samples of untreated wool, the following dyes being used—Lissamine Green VS (C.I. Acid Green 16), Neolan Green BL Conc. (C.I. Acid Green 12), and Coomassie Green TS (C.I. Acid Green 19). The conditions under which the samples were treated with THPC are summarised in Table I. In all cases the liquor:wool ratio was 100:1.

TABLE I  
Conditions of Treatment with THPC

Sample No.	Concentration of THPC solution (%)	pH <sup>†</sup>	Time	Temp. (°C)
1	0.27	2.5	66 h	R*
2	0.27	2.5	18 days	R
3	0.27	2.5	18 days	R†
4	1.9	2.5	5 h	R
5	1.9	2.5	24 h	R
6	1.9	2.5	96 h	R
7	0.74	2.5	15 min	60-65
8	0.74	5.8	4 days	R
9	0.74	7.5	6 days	R

\* R = room temp.

† Sample 3 was thoroughly rinsed in twelve successive changes of water, each rinse lasting for 10 min.

‡ pH adjustments were made by adding hydrochloric acid or 20% aq. triethanolamine solution.

In all cases, it was found that dye was preferentially absorbed by the treated samples, with the exception of sample 3, in which the prolonged rinsing had reduced the uptake to that of an untreated sample. The enhancement reaction is slow at room temperature, as shown by samples 4-6, in which a progressive deepening of shade with time of treatment was observed. If, however,

the temperature of treatment is raised, the reaction proceeds rapidly. For example, treatment for 15-20 min at 65°C (sample 7) brought about the same change as treatment for four days at room temperature (sample 6).

#### DYEING IN THE PRESENCE OF THPC

It was later found that addition of THPC to the dyebath increases the rate of uptake of acid and premetallised dyes. Much deeper shades were obtained by dyeing wool under the conditions indicated in Table II than by dyeing similar material under comparable conditions in absence of THPC. The conditions of dyeing were chosen so that in absence of THPC the uptake of dye was small, e.g. dyeing with Neolans was carried out at pH 7 and not at pH 2-3 as is more usual.

The increase in dye uptake was sometimes extremely marked. For example, a full black was obtained when Neolan Black SR Conc. was applied to samples 3(a), 3(d), and 3(e), even though in the absence of THPC the wool was coloured only pale grey. Similar effects were obtained with several dyes, but some, e.g. Irgalan Brilliant Green 3GL (C.I. Acid Green 57), could not be applied in this way, as they were precipitated. Addition of THPC to a solution of Methylene Blue (sample 5) reduced the ease with which the dye was taken up by the wool.

#### SETTING OF UNTREATED KERATIN FIBRES IN THPC SOLUTIONS

Preliminary experiments revealed that both wool and human hair may be set more readily in a 2% solution of THPC than in water of approximately the same pH. The relationships between the time of setting in the extended state and the set or supercontraction obtained are shown in Fig. 1, and it is clear that set is attained very rapidly. Because of experimental difficulties, it is not easy to detect any supercontraction which might occur in THPC solutions of pH 6.1, but there is little doubt that supercontraction would occur

TABLE II  
Conditions of Dyeing in Presence of THPC

Sample No.	Dye	Concentration of dye in solution (%)	Concentration of THPC in solution (%)	pH	Time	Temp. (°C)
1	Lissamine Green VS	0.07	0.5	2.6	17 min	70
2	Neolan Green BL Conc.	0.03	0.3	7	18 min	66
3(a)	Neolan Black SR Conc.	0.2	26.0	7	15 min	25
3(b)			1.0	0.4	15 min	B†
3(c)			0.5	1.3	10 s	B
3(d)			0.5	2.6	10 s	B
3(e)			0.5	5.1	10 s	B
3(f)			0.5	5.1	30 s	B
3(g)			3.0*	2.6	30 s	B
4(a)	Carbolan Green G 125 (C.I. Acid Green 27)	0.5	0.65	7	15 s	B
4(b)			0.5	1.3	15 s	B
4(c)			0.5	2.6	15 s	B
5	Methylene Blue (C.I. Basic Blue 9)	3.0*	2.1	7	20 s	B

\* On fabric weight.

† B = boiling point.

if the setting time were less than 1 min. Evidence in support of this was provided by the observation that extended fibres treated for 1 min in a THPC solution at pH 2.1 supercontract 13.9% when subsequently released and boiled freely in water for 1 h. Moreover, fibres set in dilute solutions of THPC supercontract, if the time of treatment is short, and these solutions also enhance the ability of the fibre to take a set (see Table III).

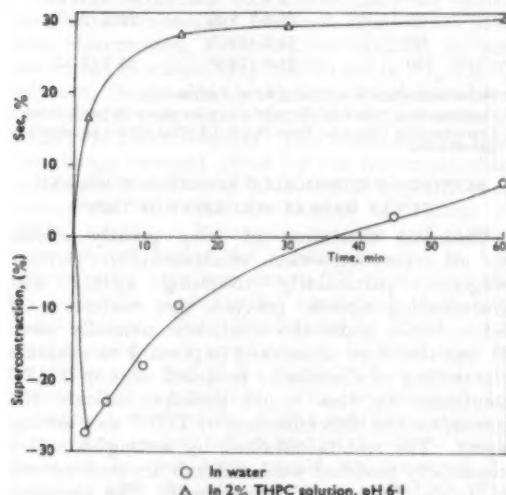


FIG. 1—Variation of set with time of treatment at the boiling point

The presence of THPC does not appear to alter the fundamental process in the supercontraction/setting mechanism, viz. that of bond fission followed by bond re-forming; the supercontraction portion of the set/supercontraction vs. setting time curve (Fig. 1) is merely compressed towards the ordinate.

The extent of the improvement in set brought about by THPC clearly will depend on, e.g., temperature, concentration, and pH. Data relating to the set or supercontraction obtained in THPC solutions under various conditions are summarised in Tables III–VI and Table VIII.

TABLE III  
Dependence of Set on THPC Concentration in Boiling Solutions

Concentration of THPC solution <sup>†</sup> (%)	% Set* after treatment in extended state for 2 min	% Set* after treatment in extended state for 120 min
0	-27.9 (-7.9†)	14.9 (14.5†)
0.005	-22.5	22.5
0.01	-26.9	31.9
0.02	-19.7	33.1
0.05	-17.3	30.0
0.07	-16.0	31.2
0.1	-17.8 (-0.5)	28.6 (29.6)
0.5	4.9 (-1.5)	22.9 (22.5)
1.0	12.9 (-2.7)	22.5 (22.9)
2.0	17.1 (-5.8)	24.5 (25.7)

\* Supercontraction is indicated by the minus sign.

† Values given in parentheses are the corresponding ones for human hair.

‡ The pH values of these solutions lie between 2.1 and 3.1.

TABLE IV  
Effect of Temperature on Setting of Wool in Water and in 2% THPC Solution\*

Temperature of setting (°C)	% Set <sup>†</sup> in water	% Set <sup>†</sup> in THPC solution (pH 2.1)
40	0.3	-10.3
50	1.5	7.1
60	-6.5	16.4
70	-15.0	22.4
80	-15.4	26.2
90	-7.8	—
100	14.9	24.4

\* The time of setting in all cases was two hours.

† Supercontraction is indicated by the minus sign.

TABLE V  
Effect of pH on Setting of Wool in Boiling 0.1% THPC Solution\*

pH	% Set <sup>†</sup> after treatment in the extended state for 2 min	% Set <sup>†</sup> after treatment in the extended state for 120 min
1.3	2.6	5.9
2.5	-17.8	28.6
3.3	-17.0	29.7
4.2	-18.4	31.2
5.4	-10.4	33.2
6.0	-3.2	31.8
8.2	-1.8	23.2
9.4	—	12.5
9.9	—	4.8
10.2	-7.7	-5.8

\* In all cases the stretched fibres were subsequently released and boiled freely in water for one hour.

† Supercontraction is indicated by the minus sign.

It seems clear from these results that THPC is effective even in very dilute solutions and that setting occurs at temperatures below 100°C. Little advantage is, in fact, gained by increasing the temperature above 70°C when 2% THPC solution is used as the setting medium. The contrast between the change brought about by water at 70°C and that caused by a 2% THPC solution at the same temperature is clearly shown in Table VI; in one case the fibres supercontract even if the time of setting is 120 min, while in the other the fibres supercontract after short times of treatment but take up set if the time of treatment exceeds 11 min.

TABLE VI  
Setting of Wool in Water and in 2% THPC Solution at 70°C

Time of setting (min)	% Set* in water	% Set* in 2% THPC solution (pH 2.1)
1	—	-0.2
2	-0.5	-0.8
5	0.8	-27.7
10	—	-9.1
15	2.6	0.0
30	-17.5	15.8
60	-25.3	19.8
120	-15.0	22.4

\* Supercontraction is indicated by the minus sign.

The effect of pH on the set obtained in THPC solution is very marked; the most favourable range seems to be pH 4–7. Strongly alkaline solutions

containing THPC are poor setting agents—thus, a set of 19.4% was obtained for wool fibres set in 6.6% triethanolamine solution, whereas fibres set in 0.1% THPC solution at the same pH (10.2) supercontract by 5.8%. This may be due to the decomposition of THPC to yield formaldehyde, since the presence of this reagent hinders setting, as shown by Table VII.

TABLE VII  
Setting of Wool in Boiling Water and in Boiling 1% Formaldehyde Solution at pH 5.5

Time of setting (min)	% Set* in water	% Set* in formaldehyde
2	— 27.9	— 28.7
30	— 6.3	— 27.2
60	— 8.4	— 26.4
120	14.9	— 26.8
240	26.3	— 21.7
480	30.9	— 10.1

\* Supercontraction is indicated by the minus sign.

Strongly acidic solutions of THPC are also less effective setting agents than neutral solutions, but the magnitude of the difference depends on the concentration of the THPC solution. For example, although a 0.1% solution of THPC at pH 1.3 imparts a set of only 5.9% after 2 hours' treatment (Table V), fibres set in a 2% solution of THPC at the same pH take a set of 25.9% in the same time (Table VIII). The ability of strongly acidic solutions of THPC to promote set is clearly shown in Table VIII.

TABLE VIII  
Setting of Wool in Boiling Strongly Acidic Solutions

Time of setting (min)	% Set* in 0.1-N hydrochloric acid (pH 1.3)†	% Set* in 2% THPC solution at pH 1.3‡
1	1.9 (— 13.2)	— 0.6 (— 1.1)
2	3.5 (— 27.9)	— 16.1 (17.1)
5	— 0.3 (— 23.2)	17.0 (19.2)
15	— 0.4 (— 9.1)	20.2 (23.5)
30	— 2.2 (— 6.3)	23.7 (24.6)
60	— 20.6 (— 8.4)	24.2 (24.7)
90	— 21.5 (14.7)	— —
120	— 21.0 (14.9)	25.9 (24.5)

\* Supercontraction is indicated by the minus sign.

† Corresponding values for fibres in water are shown in parentheses.

‡ Corresponding values for fibres in 2% THPC at pH 2.1 are shown in parentheses.

#### SETTING OF CHEMICALLY MODIFIED WOOL AND HUMAN HAIR IN SOLUTIONS OF THPC

Chemical treatment of wool greatly affects its set/supercontraction characteristics. Several reagents, particularly oxidising agents and deaminating agents, prevent the realisation of set in water under the conditions normally used. It was therefore considered important to examine the setting of chemically modified wool in THPC solutions, for this would perhaps indicate the reason for the high efficiency of THPC as a setting agent. The results obtained by setting extended chemically modified wool and hair are summarised in Tables IX and X, respectively. The chemical treatments used to modify the fibres are those given on page 193.

TABLE IX  
Setting of Chemically Modified Wool in Boiling 2% THPC Solution (pH 2.1)

Treatment	% Set* obtained after setting in the stretched state for					
	1 min	2 min	5 min	15 min	30 min	60 min
Deamination	— 8.1 (— 0.6)	— 20.7 (— 1.0)	— 6.0 (— 1.7)	14.8 (— 3.4)	17.8 (— 3.5)	18.5 (— 3.8)
	— 13.3 (— 0.6)	18.1 (— 1.3)	24.5 (— 0.5)	25.9 (— 1.4)	27.6 (— 1.5)	28.1 (— 1.3)
Iodination	— 13.3 (— 0.6)	18.1 (— 1.3)	24.5 (— 0.5)	25.9 (— 1.4)	27.6 (— 1.5)	28.1 (— 1.3)
	— 6.9 (— 1.9)	20.6 (— 2.7)	21.1 (— 8.4)	20.7 (— 5.8)	22.2 (— 1.1)	21.6 (— 6.5)
Permanganate oxidation	— 24.3 (— 30.1)	— 3.1 (— 30.4)	9.0 (— 23.1)	7.6 (— 18.5)	9.8 (— 8.9)	14.2 (— 3.0)
	— 2.0 (— 0.9)	18.9 (— 0.9)	21.4 (— 4.0)	22.0 (— 9.1)	21.3 (— 9.9)	21.5 (— 3.8)
Peracetic acid oxidation	— 2.0 (— 0.9)	18.9 (— 0.9)	21.4 (— 4.0)	22.0 (— 9.1)	21.3 (— 9.9)	21.5 (— 3.8)

\* Supercontraction is indicated by the minus sign. The corresponding values for the treated fibres set in water are shown in parentheses.

TABLE X  
Setting of Chemically Modified Hair in Boiling 2% THPC Solution (pH 2.1)

Treatment	% Set* obtained after setting in the stretched state for					
	2 min	5 min	15 min	30 min	60 min	120 min
Deamination	— 2.2 (— 1.0)	— 6.1 (— 1.1)	10.1 (— 2.1)	14.1 (— 2.0)	17.5 (— 4.0)	20.2 (— 1.1)
	— 5.5 —	20.6 (— 3.5)	23.6 (— 5.7)	26.1 (— 4.4)	26.6 (— 2.4)	25.0 (— 3.4)
Hydrogen peroxide	— 3.9 (— 0.4)	5.9 (— 0.8)	17.2 (— 1.0)	21.2 (— 2.3)	20.6 (— 0.6)	15.9 (— 4.4)
	— 11.8 (— 0.9)	— 5.8 (— 1.8)	— 1.7 (— 3.0)	5.2 (— 5.2)	10.5 (— 6.7)	13.8 (— 3.9)
Potassium cyanide†	— 3.8 (— 1.3)	— 10.1 (— 1.1)	— 11.0 (— 1.1)	— 12.5 (— 1.6)	— 12.1 (— 2.7)	— 11.8 (— 3.8)
	— 2.0 (— 0.9)	18.9 (— 0.9)	21.4 (— 4.0)	22.0 (— 9.1)	21.3 (— 9.9)	21.5 (— 3.8)

\* Supercontraction is indicated by the minus sign. The corresponding values for the treated fibres set in water are shown in parentheses.

† Fibres treated with potassium cyanide were extended only 30%.

TABLE XI  
Setting of Wool and Human Hair in Reagents related to THPC

Fibre	Reagent	% Set* realised after 2 min	% Set* realised after 120 min
Wool	Cetyltrimethylammonium bromide	— 10.8 (— 27.9)	— 2.6 (14.9)
Wool	Tributylhydroxymethylphosphonium chloride	— 0.6	19.4
Hair	Tetrakis(hydroxymethyl)phosphonium acetate	16.1 (— 7.9)	33.4 (14.5)
Hair	Tris(hydroxymethyl)phosphine oxide	— 0.7	10.1

\* Supercontraction is indicated by the minus sign. The corresponding values for fibres set in water are shown in parentheses.

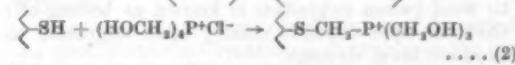
Several important conclusions may be drawn from the above results. Although fibres which have been deaminated, iodinated, or oxidised do not take a set in water, they can be set in 2% THPC solution. On the other hand, fibres which have been treated with potassium cyanide do not set either in water or in THPC solution. This would suggest that the change brought about by the treatment with potassium cyanide is of fundamental importance in connection with the mechanism of setting in THPC solutions. Potassium cyanide and caustic soda applied under the conditions used in the present work have been shown<sup>9-11</sup> to stabilise the  $-S-S-$  bonds, probably by converting them into  $C-S-C$  bonds. The effectiveness of THPC as a setting agent may consequently be attributed to its action on the  $-S-S-$  bonds, the most likely process being reduction. This is followed by oxidation to rebuild the  $-S-S-$  bonds in the strained state. Any reagent which makes the  $-S-S-$  bonds resistant to reduction would be expected to interfere with the setting process in THPC. The effectiveness of THPC solution in setting deaminated, iodinated, or oxidised fibres is no doubt associated with the complexity of the reactions taking place. Fibres which have been deaminated or iodinated can be set because the main rebuilding in THPC setting probably occurs by reoxidation of bonds reduced by THPC. Neither the amino groups nor the tyrosine residues are greatly concerned. Their modification may hinder the setting process but will not prevent it. The oxidising agents probably bring about limited conversion of the  $-S-S-$  bonds to stable units, such as  $-SO_3$  groups. Much of the change will, however, be limited to the formation of intermediates which can be reduced, first to  $-S-S-$  bonds by the THPC, these bonds then being broken and subsequently rebuilt in the usual manner. The role of the  $-S-S-$  bonds is not, however, a simple one. Some confirmation of the view that the usefulness of THPC as a setting agent is connected with its reducing properties was obtained by examining the set/supercontraction characteristics of fibres in the following related compounds—cetyltrimethylammonium bromide, tetrakis(hydroxymethyl)phosphonium acetate, tris(hydroxymethyl)phosphine oxide, and tributylhydroxymethylphosphonium chloride. The results obtained are summarised in Table XI.

Tetrakis(hydroxymethyl)phosphonium acetate is by far the most effective; it facilitates setting more than does THPC. Tris(hydroxymethyl)phosphine oxide, on the other hand, does not enhance set, presumably because it is comparatively stable and has little reducing power. The charge on the reagent is not, apparently, a

critical feature, since cetyltrimethylammonium bromide does not enhance set.

### Conclusions

It would appear from the foregoing discussion that reduction is the main reaction between THPC and wool. This would account for the high degree of set obtained in solutions of THPC and for the failure of fibres treated with potassium cyanide to take a set in THPC solutions, even though fibres chemically modified by deamination, iodination, or oxidation can be set in this medium. Account must, however, be taken of the dyeing characteristics of THPC-treated wool. Some of the enhanced dye uptake is undoubtedly due to the greater accessibility of the treated fibres consequent upon bond fission. It is likely, however, that treatment with THPC also introduces charged groups into the fibre, for while the take-up of acid dyes is increased, the absorption of basic dyes falls. This suggests that two reactions are taking place, (a) a fission, probably reduction of the  $-S-S-$  bonds (Eqn. 1), and (b) an interaction with the THPC involving the products of the fission of the  $-S-S-$  bonds and other groups in the keratin molecule, e.g.  $-NH_2$  and  $-OH$ , resulting in the introduction of positive charges, as in Eqn. 2.



The reactions taking place are probably very complex, for THPC is highly reactive; further work is in progress to elucidate the details of the reactions.

\* \* \*

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## Foam, Flow, and Level Dyeing

### Some New Information on the Factors Influencing the Flow of Dye Liquor through Wool Packages

H. R. HADFIELD and D. R. LEMIN

Many auxiliary products encourage the production of foam in the dyebath. A quantitative study has been made of the effect of this foam on the flow of dye liquor through a textile package. It is shown that a build-up of foam can almost stop dye-liquor flow. Silicone antifoaming agents are shown to be very valuable in eliminating the deleterious effect of foam. The method of using this type of product is very important if the full effect is to be obtained. The mechanical properties of the foam are sufficient to explain the behaviour observed.

#### Introduction

In dyeing any textile material it is desirable that the dye liquor is uniformly distributed over and throughout the material. Except in the special case of padding, the dyeing systems employed in practice do not allow the almost instantaneous, uniform presentation of the dye liquor, in a practically unchanged condition, to the textile material. In the majority of dyeing systems, level dyeing is achieved as a result of the cumulative effect of four main properties of the system. These are listed below and their modes of action briefly described.

*Migration of dye* This property of the dye-fibre system allows for the eventual production of a level dyeing even though the initial absorption of dye has been non-uniform. The use of this property by the dyer is decreasing as a result of the introduction of more efficient dyeing machines and the use of dyes of better wet fastness (which generally have poor migrating properties). It is in the application of direct dyes to cotton and level-dyeing acid dyes to wool (when migration is known as boiling-off) that the dyer depends mainly upon migration to produce level dyeings.

*Slow rate of initial absorption of dye* This is sometimes known as a "slow strike". For any given dyebath exhaustion, the integration of rate of dyeing properties and rate of migration properties of a dye-fibre system governs the level-dyeing properties of the system. The slower the rate of dyeing the more uniform will be the initial dye exhaustion and, as a result, less migration of dye will be needed to produce a level dyeing. The introduction of fast dyes has resulted in an increased need to control carefully the rate of dye absorption.

*Exhaustion of dye* In all types of package dyeing, a high dyebath exhaustion increases the difficulty of producing level dyeings. The qualitative experience of practical dyers has received quantitative confirmation by Boulton and Crank<sup>1</sup>, who demonstrated mathematically that the time, or alternatively the rate of flow of dye liquor, required to produce a level dyeing increased as the exhaustion of dye increased. Generally, and particularly in wool dyeing, exhaustion is not a variable readily changed by the dyer and, in the range of exhaustions generally acceptable to the wool dyer (70-95%), a change in exhaustion does

not, according to these workers, greatly affect the ease of producing level dyeings.

*Distribution of dye liquor over the textile material* In the special cases of package, beam, and hank dyeing this is governed by the rate of circulation of dye liquor through the material. The other systems, which are not of primary concern in this paper, are jig and winch dyeing. In a general way the relation between rate of circulation of dye liquor and the other properties contributing to level dyeing can very readily be seen. If the dye liquor could be presented completely uniformly to the textile material, then level dyeing would be assured without the need for slow exhaustion or good migration. The other extreme is the case of very poor dye liquor circulation, where, no matter how slow the exhaustion, the absorption of dye will be non-uniform and hence level dyeing can be achieved only by subsequent migration. Thus, with really poor dye liquor circulation, uniform application of the non-migrating type of dye is virtually impossible. Boulton and Crank<sup>1</sup> have, by a mathematical treatment, convincingly demonstrated the importance of dye liquor flow in package dyeing.

A tremendous amount of work carried out by dye manufacturers, the Society of Dyers and Colourists, and other organisations on the level-dyeing properties of dyes has resulted in the production of manuals and pattern cards containing assessments and graphical representations of the dyeing properties of dyes. Valuable though these results are, it must be realised that, unless the dye liquor is allowed to flow through the textile material, level dyeing is virtually impossible. The practical dyer, drawing on his experience, is able to make suitable modifications to the recommended procedures, according to the dyeing system he is using, but, in spite of this, results far different from those expected are sometimes obtained and often the cause can only be poor circulation of dye liquor through the package.

A study has been made of some of the factors affecting the rate of circulation of dye liquor through a textile package. It has been concerned with the average flow over the whole package and no attempt has been made to study the important, but secondary, effect of the distribution of flow within the package. Some recent work on this aspect of liquor flow uses a method of analysis by electrical analogy<sup>2</sup>.

### General Principles

The flow of a fluid through a porous bed at relatively low speeds, e.g. the flow of dye liquor through textile packages, obeys Darcy's law—

$$Q = K \Delta P / L \quad \dots \dots (1)$$

where  $Q$  is the flow for unit cross-section,  $K$  is a constant,  $\Delta P$  is the pressure difference across the porous bed, and  $L$  is the thickness of the package.

$K$  can be written as  $K = k/\mu$ , where  $k$  is a new constant and  $\mu$  is the viscosity of the liquid.

Therefore,

$$Q = k \Delta P / \mu L \quad \dots \dots (2)$$

It can also be shown, using Hagen-Poiseuille's Law, that, for an idealised package consisting of parallel channels arranged in the direction of flow of the liquid,

$$k = n \pi d^4 / 128 \quad \dots \dots (3)$$

where  $d$  is the diameter of the channels, and  $n$  is the number of the channels per unit cross-section.

The above equations are interesting to the dyer although they add nothing new to his information. The significance of the quantities in Eqn. (2) and (3) is as follows—

$\Delta P$  The rate of flow increases as the pressure increases. However, textile packages are not rigid, and too high a pressure can change the dimensions of the packages, effectively reducing  $d$  and thereby reducing the flow, which varies as the fourth power of  $d$ . There is clearly an optimum pressure for maximum flow.

$L$  As the thickness of the package increases the flow decreases.

$\mu$  At constant pressure, the flow of liquor through a package is increased as the temperature is increased owing to the reduction, with increase in temperature, of the viscosity of the dye liquor.

Increased circulation with increase in temperature assumes a constant pressure from the pumps; as the temperature approaches 100°C this is not achieved in a dyeing machine owing to "pump slippage", unless the machine is pressurised to prevent boiling in the pump.

$d$  Eqn. (2) and (3) show that the flow of liquid increases as the fourth power of the diameter of the channels. The possibility of  $d$  being decreased as a result of high circulation pressures has already been mentioned. The greater ease of production of level dyeings on coarse yarns, where the channels are wider, than on fine yarns can readily be understood.

### The Role of Foam in the Dyebath

The problems arising from the production of foam in a dyeing machine are not clearly understood. The practical dyer has learnt, through hard experience, that in certain systems the presence of foam can lead to unsatisfactory dyeings, but in general this experience is mainly restricted to the difficulties caused by flotation of yarn in a Hussong machine or of lightweight fabrics on a winch. Certain experiences gained in developing practical dyeing methods for Terylene led to the belief that foam formation could be a

critical factor in preventing the production of level dyeings.

**Example 1** When Terylene slubbing was dyed in a traditional Callebaut De Blicquy eight-can dyeing machine, level results were obtained without difficulty. Shortly afterwards, on exactly the same type of machine, but in another dyehouse, very unlevel results were obtained on an identical dyeing. The pump efficiency was unaltered, and the cause was ultimately found to be a leaking gland which allowed air to be sucked into the dyeing system. In the presence of dispersing agent the air produced foam and the foam prevented circulation of dye liquor. An immediate solution was provided by adding some antifoaming agent, but a subsequent, more satisfactory, solution was to mend the leaking gland.

**Example 2** Attempts have been made to dye Terylene slubbing in a one-stick Hussong machine. Good circulation through the material was obtained until the temperature approached the boiling point. At this stage, circulation through the material stopped completely, owing to the formation of foam. The steam caused foam to form as it no longer condensed quickly in the very hot water. Addition of antifoaming agent produced a marked improvement, but this dyeing system never proved completely satisfactory.

**Example 3** Laboratory tests showed that Lissapol N improved the level-dyeing behaviour of many acid dyes on wool, but in practical dyeing the results obtained in the presence of Lissapol N were often inferior to those obtained in its absence, even though laboratory tests indicated that the agent should produce a significant improvement in level dyeing. The cause of this discrepancy was again thought to be the production of foam in the dyebath.

In spite of many cases similar to those described, no quantitative information is available on the role of foam in the dyeing of textiles. In view of the very considerable importance of the subject, it was decided to investigate the subject more fully.

### Experimental Method

After preliminary trials, an apparatus was constructed which is shown diagrammatically in Fig. 1.

Water from a tank is passed through a Stuart Turner No. 1 centrifugal pump capable of delivering 2 gal/min at a 5-ft head. A pressure check showed that the pump was capable of delivering water at a pressure of 12 lb/in<sup>2</sup>. The flow from the pump was then split by means of a three-way tap so that part went through a by-pass back to the reserve tank and the rest into the base of a glass tube, 2 ft 6 in. long and approx. 2 in. in diameter. This tube was fitted with a sintered glass plate at its base through which compressed air could be passed. The tube was fitted with an overflow tube 9 in. from the top. The liquid pumped into the tube, after passing upwards through it, returned to the reserve tank by way of the overflow. The material used as a plug—20 discs of botany serge, of radius 2.4 cm—was clamped between two perforated stainless-steel

plates and, after being wetted in boiling water, was forced into the tube and clamped so that the top plate was 3 in. below the level of the overflow.

The three-way tap was adjusted so that the flow through the tube in absence of wool was 2300 ml/min. The packing of the wool was adjusted to give a flow of approx. 800–900 ml/min and a pump pressure of approx. 4 lb/in<sup>2</sup>.

The purpose of the sintered glass plate and compressed air was to produce foam, if required, in the tube below the package. The purpose of the three-way tap at A was to provide a practically constant pressure to circulate the dye liquor. Thus, a change in the resistance to flow at W would result in a marked reduction in the volume flowing through W and an increase in the volume flowing through tube O, so that there would not be a very marked increase in pressure which could cause deformation (mainly compression) of the package W. This device helps to give reproducible results and also increases the sensitivity of the apparatus, so that small changes in the resistance to flow at W result in a larger change in rate of flow through W than would be obtained in a commercial dyeing machine.

The complete apparatus is shown in Fig. 2. The method of operation was as follows. The wetted-out material was entered into the tube and clamped. Distilled water (6 l.) was put in the reserve tank, the pump started and the liquor circulated for 20 min, after which the flow was measured by running the liquor from H into a measuring cylinder. This gave the "water control" figure.

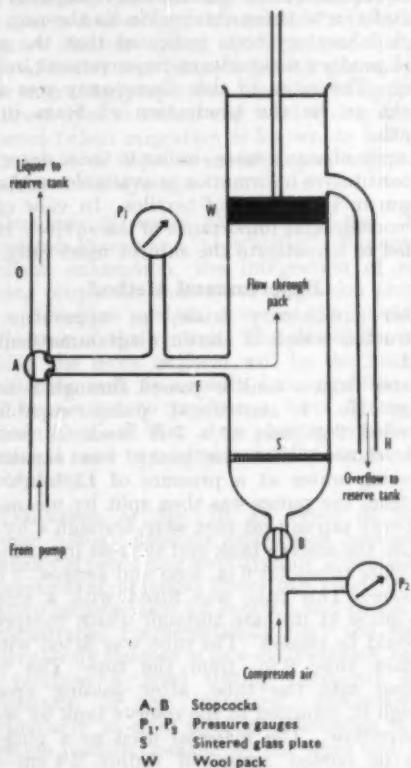


FIG. 1

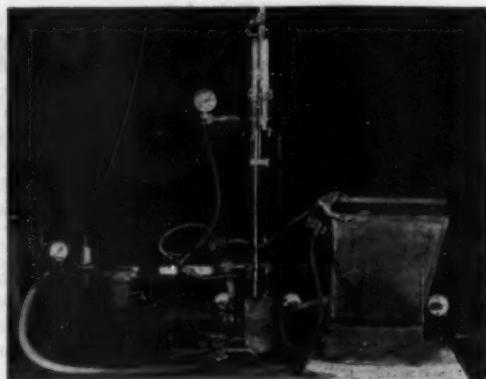


FIG. 2

The acid (or alkali) was then added and circulated for 20 min, when a further flow reading was taken. The agent or dye under examination was then added and the liquor again circulated for 20 min to attain equilibrium before again measuring the flow. Air could be injected at any stage of the process, being passed through a metering orifice 0.2-mm in diameter, at a pressure of 2 lb/in<sup>2</sup> above that given by the pump, before entering the system through the perforated glass plate (porosity 4)\* at the base of the glass tube. The metering jet and pressure ensured that the volume of air was constant, and the glass plate assisted the formation of bubbles of uniform size. All experiments were carried out in duplicate, at least. Reproducible packages, with equal resistance to flow, could be obtained by tightening or slackening the screw holding the cloth discs between the perforated plates.

### Results of Flow Experiments

#### PRELIMINARY RESULTS ON WOOL AND OTHER FIBRES

The first experiments were carried out on wool at a liquor temperature of 20°C. The 20 discs were of Botany serge (6 oz, 2/2 twill, 60 picks/in., 70 ends/in.) and the liquor contained 3 g/l. sulphuric acid in distilled water. The rate of flow through the control was approx. 900 ml/min. The results obtained are given in Table I. The tests were carried out under acidic conditions, as this resulted in a more stable package and also eliminated any foaming resulting from impurities or decomposition products being removed from the wool.

The results show that, as expected, the presence of certain auxiliary products and air in the circulating system prevents, almost completely, the flow of liquid through the wool package. No foaming occurred under conditions of good flow, whereas there was a marked build-up of foam below the package when the flow was almost completely stopped. There is little doubt that the results detailed in Table I exaggerate the effect, since the apparatus was designed to do this. Nevertheless, it is considered that changes of the order of 30% or more will certainly be significant in practical dyeing.

\* BS 1752:1952 Sintered disk filters for laboratory use.

TABLE I

Conditions	Flow (%)
No auxiliary product	100 (control)
No auxiliary product + air stream	84
3 g/l. Lissapol N <sup>a</sup>	92
3 g/l. Lissapol N + air stream	2.2
1 g/l. Lubrol W <sup>b</sup>	85
1 g/l. Lubrol W + air stream	4.6
1 g/l. Lissolamine A 50% <sup>c</sup>	90.0
1 g/l. Lissolamine A 50% + air stream	1.9
1 g/l. Carbolan Salt A <sup>d</sup>	98
1 g/l. Carbolan Salt A + air stream	74
1 g/l. Dispersol AC <sup>e</sup>	89
1 g/l. Dispersol AC + air stream	37
3 g/l. Lissapol D <sup>f</sup>	98
3 g/l. Lissapol D + air stream	1.4
3 g/l. Dispersol CWL <sup>g</sup>	86
3 g/l. Dispersol CWL + air stream	11

<sup>a</sup> Lissapol N alkylphenol condensed with ethylene oxide.  
<sup>b</sup> Lubrol W fatty alcohol-ethylene oxide condensate.  
<sup>c</sup> Lissolamine A cetyltrimethylammonium bromide.  
<sup>d</sup> Carbolan Salt A sodium salt of a naphthalene disulphonate acid.  
<sup>e</sup> Dispersol AC naphthalene formaldehyde sulphonate acid.  
<sup>f</sup> Lissapol D cetyl-oleyl sodium sulphate.  
<sup>g</sup> Dispersol CWL fatty acid amine condensed with ethylene oxide.

The effect of foam in reducing the circulation of dye liquor through packages is not restricted to flow through wool. Table II details results obtained on viscose rayon and on Terylene. The temperature was again 20°C and the liquor contained 3 g/l. sulphuric acid.

TABLE II

Conditions	Flow (%)		
	Wool*	Viscose rayon†	Terylene‡
	100 (control)	100 (control)	100 (control)
No auxiliary product			
No auxiliary product + air stream	84	49	55
3 g/l. Lissapol N	92	100	100
3 g/l. Lissapol N + air stream	2.2	2.0	0

\* Botany serge, 6 oz, 2/2 twill, 60 picks/in., 70 ends/in., 20 discs.

† Plain weave, spun yarn, 9½ oz, 25 picks/in., 30 ends/in., 20 discs.

‡ Plain weave, spun yarn, 4½ oz, 50 picks/in., 50 ends/in., 100 discs.

With viscose rayon and Terylene, air alone produces a bigger reduction in flow than with wool, but the effect of foaming conditions is also very marked indeed; with Terylene, flow of liquor is completely prevented.

#### EFFECT OF TEMPERATURE

Preliminary experiments were carried out at 20°C, mainly for convenience. It is obviously desirable to confirm that the behaviour found at 20°C is maintained as the temperature is increased. Tests were therefore carried out at 60 and 80°C; the results are listed in Table III. The liquor conditions and the wool serge were as before. Temperatures higher than 80°C were not used because "pump-slippage" led to difficulties in the reproduction of results. Although it would have been preferable to examine the behaviour at 95–100°C, it is considered that, if there is no change in pattern of behaviour at 20, 60, and 80°C, it is reasonable to assume that there will not be any sudden changes between 80 and 95°C.

TABLE III

Conditions	Flow (%) at		
	20°C	60°C	80°C
No auxiliary product	100 (control)	100 (control)	100 (control)
No auxiliary product + air stream	84	86	84
3 g/l. Dispersol CWL	86	79	64
3 g/l. Dispersol CWL + air stream	11	4.3	2.2
1 g/l. Carbolan Salt A	98	83	80
1 g/l. Carbolan Salt A + air stream	74	50	21

The results given in Table III confirm the earlier ones, which show the very serious effect of foam on the circulation of dye liquor. Carbolan Salt A, which does not, in the presence of air, markedly reduce the circulation at 20°C, has an increased effect as the temperature is raised. Even at 80°C, however, the flow in presence of Carbolan Salt A is still ten times higher than that in presence of Dispersol CWL.

#### EFFECT OF ANTIFOAMING AGENTS

Many agents are available which prevent the formation of a stable foam. It is obviously of interest to determine whether the addition of these agents reduces the deleterious effect of certain auxiliary products when used under conditions which give rise to foaming. The results of such an investigation are given in Table IV. The liquor was distilled water (pH 7) at 80°C and the wool package was identical with that previously used.

TABLE IV

Conditions	Flow (%)	
	100 (control)	100 (control)
No auxiliary product		
3 g/l. Dispersol CWL	94	
3 g/l. Dispersol CWL + air stream	3.0	
3 g/l. Dispersol CWL + air stream + 20 parts/million Silicone Antifoam M 435 (added after formation of foam)	26.2	

The results show that the addition of Silicone Antifoam M 435 had a marked effect. The circulation is still low compared with that in absence of an air stream, but the important point is that the circulation is increased almost nine times by the addition of the agent. It is considered that this increase is very significant, and that it is appropriate to suggest that, when agents are used which are known to produce foam, the addition of an antifoaming agent should be standard practice. In interpreting the results it is important to remember that the apparatus used was designed to emphasise foaming behaviour; in practical dyeing the conditions for foam formation will rarely be as good as in the experimental apparatus, and the rate of circulation will not reflect so sensitively the resistance to flow caused by the build-up of foam. In practical tests Dispersol CWL used under foaming conditions gave poor results, but with the correct use of an antifoaming agent the expected improvement based on laboratory-scale dyeing tests was obtained, indicating that the circulation was largely unaffected.

Too much stress cannot be laid on the correct use of the antifoaming agent. In the tests described, if the antifoam was added to the reserve tank, no positive effect was observed. This suggests that highly insoluble antifoaming agents serve no useful purpose if put in the reserve tank. The positive results recorded in Table IV were obtained by injecting the agent at F (Fig. 1) so that it was forced through the wool package. In practical dyeing similar precautions must be taken. The antifoaming agent must be added before the Dispersol CWL (or other agent which could cause foaming) in such a manner that it is distributed through the package. Further information on the addition of antifoaming agent is given later in the paper. Organic antifoaming agents also give beneficial effects, but suffer from the defect of loss by evaporation. As they are to some extent water-soluble, the stage and point of addition are not as critical as with the highly insoluble silicone antifoaming agents.

No improvement in circulation was obtained by increasing the concentration of Silicone Antifoam M 435 from 67 to 200 parts/million.

It has been found that not all antifoaming agents are suitable, particularly for package dyeing. In one experiment with a 10% emulsion of silicone oil (as distinct from a 30-35% emulsion of silicone oil, as in Silicone Antifoam M 437), a flocculant white precipitate was deposited on the package. This deposit has, of course, made the agent unacceptable. A deposit was formed because the emulsifying agent used is not compatible with the dyes or auxiliary products in the dyebath. Difficulties of this type will probably prove more common with 10% emulsions than with more concentrated ones, because of the greater difficulty in preparing dilute emulsions that are stable to prolonged storage.

#### EFFECT OF DYEBAATH pH

The wool dyer is well aware that the pH of the dye liquor can influence its rate of flow through a wool package. The effect of foaming conditions over a range of dyebath pH has been investigated at 60°C; the results are given in Table V. The wool package was the same as that previously used.

TABLE V

Conditions	Acid 3 g/l. $H_2SO_4$	Flow (%) Neutral pH 7 (NaOH, citric acid)	Alkaline 3 g/l. $Na_2CO_3$
No auxiliary product	86	100 (control)	40
No auxiliary product + air stream	70	39	—
3 g/l. Dispersol CWL	79	94	31
3 g/l. Dispersol CWL + air stream	4.3	7.0	1.3
1 g/l. Carbolan Salt A	83	11	29
1 g/l. Carbolan Salt A + air stream	50	13	1.4

In a general way the results in Table V indicate that foam-producing conditions have similar effects whether dyeing takes place in acid, neutral, or alkaline dyebaths. The results also show that

under neutral or alkaline conditions in absence of an auxiliary product, poor circulation may be obtained. Under alkaline conditions the poor circulation is obtained without the air stream and this is thought to be due to the increase in plasticity of wool, resulting in compression of the package and a reduction in the channel size. The poor flow obtained under neutral conditions in absence of auxiliary product or in presence of Carbolan Salt A and an air stream is difficult to explain. It may be caused by foam-producing compounds removed from the wool.

#### EXPERIMENTS UNDER CONDITIONS CLOSER TO PRACTICAL DYEING

All the experiments on liquor flow so far described were carried out with the apparatus arranged to give a virtually constant liquor pressure. The reason for this has already been explained. Nevertheless, it would clearly be of interest to carry out experiments in which the by-pass at A (Fig. 1) was removed and therefore any change in resistance to flow at the package would affect both the quantity of dye liquor flowing and the pressure at the positive side of the pump. Such experiments have been done and the results obtained are given in Tables VI and VII. The results in Table VI were obtained under neutral conditions at 20°C, and those in Table VII in presence of 3 g/l. sulphuric acid at 80°C. Acidic conditions were used for the tests at 80°C because previous experiments, already described, indicated that neutral conditions at this temperature gave less conclusive results owing, it is thought, to some decomposition of the wool.

TABLE VI

Conditions	Circulation pressure (lb/in <sup>2</sup> )	Flow (%)
No auxiliary product distilled water	8.0	100 (control)
1 g/l. Lissolamine A 50%	8.5	100
1 g/l. Lissolamine A 50% + air stream	12	11
1 g/l. Lissolamine A 50% + air stream + Antifoam M 437* added after foam formation	10.5	46

\* Added in the proportion 1 oz/100 gall, as recommended for practical use.

TABLE VII

Conditions	Circulation pressure (lb/in <sup>2</sup> )	Flow (%)
No auxiliary product	7.5	100 (control)
1 g/l. Lissolamine A 50%	7.5	90
1 g/l. Lissolamine A 50% + air stream	9.5	29
1 g/l. Lissolamine A 50% + air stream + Antifoam M 437 added after foam formation	8	50

The results show that the pattern of behaviour is maintained. Foam causes an increase in circulation pressure and a marked decrease in the flow of liquor through the package. Addition of antifoam

is again advantageous, although the improvement is greater at 20°C than at 80°C. At both 80°C and 20°C addition of antifoam does not restore the flow to 100%. It must be remembered that the apparatus used employs a continuous air stream. Under the less severe conditions more likely to be found in practice, the effect of foam will probably be less marked and, therefore, circulation in presence of an antifoaming agent will not be as seriously affected.

It was reasoned that addition of antifoaming agent after the formation of foam was unsound practice for, if the circulation is reduced by the presence of foam, and the foam is stable in the textile package, it is possible that the antifoaming agent will never get completely through the package. Under these conditions it would not be surprising if the full beneficial effect of the antifoaming agent was not obtained. To test if this was, in fact, the cause of the failure of antifoaming agent to restore circulation to 70–100%, experiments were carried out in which Antifoam M 437 was added at the outset, before the addition of Lissolamine A and the use of an air stream. The results obtained are given in Table VIII, the conditions being the same as those of Table VII.

TABLE VIII

Conditions	Circulation pressure (lb/in <sup>2</sup> )	Flow (%)
No auxiliary product	7.0	100 (control)
Silicone Antifoam M 437 (1 oz/100 gall)	7.0	100
Silicone Antifoam M 437 + 1 g/l. Lissolamine A 50%	8.0	90
Silicone Antifoam M 437 + 1 g/l. Lissolamine A 50% + air stream	8.0	79

It is considered that the results in Table VIII, when compared with those in Table VII, are of the greatest practical importance. If antifoam is added after the formation of the foam only a partial effect is obtained, but if it is added before foaming can take place, a much more pronounced effect on flow is obtained.

A further example of the importance of avoiding foam in the dyebath was found in dyeing trials with Coomassie Fast Grey 3G (C.I. Acid Black 48) on wool cheeses. A series of experiments had shown that addition of an experimental auxiliary product retarded the absorption of this dye at 80°C. It was therefore expected that when the dye was applied in presence of the experimental auxiliary product, dyeing being commenced at 80°C, level dyeings would prove more easy to obtain. Dyeing experiments on single cheeses of worsted yarn showed, in fact, that the reverse was true. All possibility of dye precipitation was eliminated and it could only be concluded that foaming, preventing circulation of dye liquor, was the cause. A further series of dyeings was carried out with the auxiliary product, but using air-free water and circulating the antifoaming agent through the package for 30 min before adding dye. This resulted in the production of very level dyeings. One is tempted to wonder

how many experiments have been wrongly interpreted, in the works and in the laboratory, as a result of the flow of dye liquor through the package being prevented by formation of foam.

#### EFFECT OF DYE

Many dyes used for dyeing wool resemble those auxiliary products which facilitate the production of foam; Carbolan Crimson B (C.I. Acid Red 138) is a well-known example. Tests have confirmed that Carbolan Crimson B in the dye liquor, in the presence of air, produces a stable foam which very seriously reduces the circulation of dye liquor. Other dyes which possess surface-active properties also showed a positive effect. The precautions necessary when using dyes of this type have been clearly indicated.

#### Flotation Experiments

It will have become apparent that the phenomenon described above is closely connected with flotation. The relationship has been studied experimentally on a small scale with 8 lb of wool (4/19s hand-knitting yarn, 64s quality) in a single-stick laboratory-scale Hussong dyeing machine. The extent of flotation was measured by

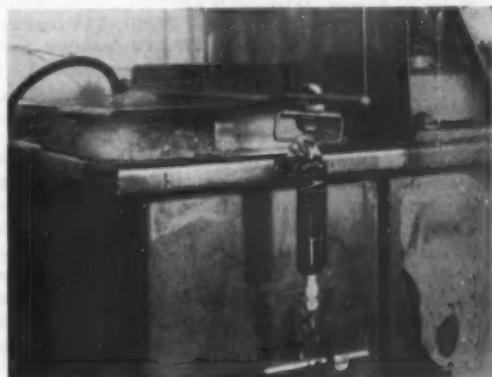


FIG. 3

attaching two spring balances, one to each side of the yarn holder, in such a manner that any change in the upthrust on the holder (when the circulation was from bottom to top) was recorded on the balances. In addition, the effect of foaming conditions was determined by introducing an air stream at the bottom of the machine, directly beneath the load. The arrangement of the apparatus can be seen in Fig. 3.

TABLE IX

Conditions	Total upthrust on cage (kg)
Water only	16
Water + 3 g/l. Dispersol CWL	16
Water only + air stream	17
Water + 3 g/l. Dispersol CWL + air stream	27.2
Water + 3 g/l. Dispersol CWL + air stream + Silicone Antifoam M 437 (1 oz/100 gall)	25.4

The results of a study of flotation, using the method described above, are given in Table IX. Experiments were carried out at 20°C and pH 7, and the liquor capacity of the machine was 140 l.

Similar results to those given in Table IX were obtained with other agents which were known to produce a stable foam. Examination of the results in Table IX shows that the impermeable nature of the stable foam produced is reflected in the very large increase in the upthrust when foaming conditions (air + agent) are used. The effect of Antifoam M 437 is positive but not large, and the lack of a greater effect is, perhaps, disappointing. In the experiment, however, the agent was added after the reading under foaming conditions had been obtained, and it was again thought that the liquor was unable to displace the foam from the package completely and thereby restore normal circulation, which would result in a lower resistance.

The effect of adding the Silicone Antifoam M 437 before the production of foam-forming conditions is detailed in Table X, the conditions being the same as in Table IX.

TABLE X

Conditions	Total upthrust on cage (kg)
Water only	16
Water + Silicone Antifoam M 437 (1 oz/100 gall)	16
Water + Silicone Antifoam M 437 + 3 g/l. Dispersol CWL	16
Water + Silicone Antifoam M 437 + 3 g/l. Dispersol CWL + air stream	18

Comparison of these results with those of Table IX again forcibly emphasises the need to prevent the formation of foam rather than to try and eliminate foam after it has been formed. It is for this reason that Antifoam M 437 should be added before foaming conditions are produced. To be safe, this means that the antifoam should be the first addition to the dyebath and should be allowed to circulate before the dye and dyeing assistants are added.

Almost identical results to those recorded for Dispersol CWL were obtained with Lissapol N, and it is believed that the behaviour is general. In a commercial dyeing on a 500-lb single-stick yarn-dyeing machine, further confirmation of this behaviour has been obtained<sup>2</sup>. Under non-foaming conditions at 418 volts, the motor consumed power at the rate of 2.2-2.6 amps, whilst under foaming conditions the consumption rose to 3.9-5.8 amps.

Practical hank-dyeing experiments have shown that certain agents which assist level dyeing of wool and which themselves foam may leave the yarn in a poor condition, suggesting that milling has taken place. The explanation for this behaviour is not known. It has however been conclusively demonstrated in practical dyeing tests that the correct use of a compatible antifoaming agent completely eliminates this effect which, in the case of fine-quality wool yarns, can be very serious.

#### Properties of Foam

The question posed by the results presented is—how does foam prevent the ready flow of dye liquor

through a package? The viscosity of the foam produced in the experiments described has been measured with a Brookfield viscometer and the results are presented in Fig. 4. Clearly, the foam

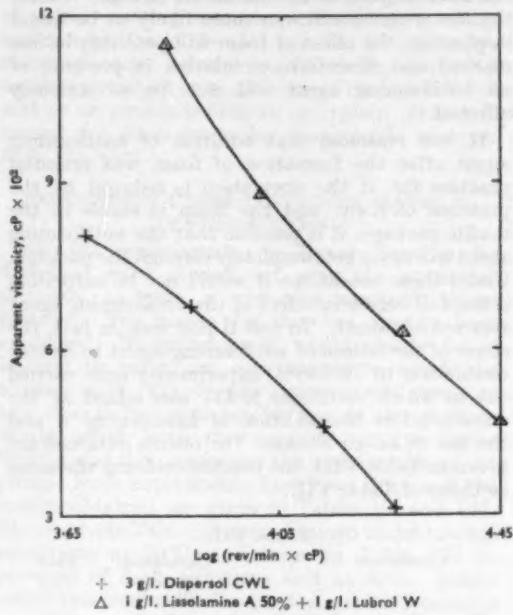


FIG. 4

will not behave as a Newtonian fluid and the viscosity will fall as the applied shear rate is increased. Nevertheless, if the lowest value of the viscosity is taken as 310 cP (310 times that of water), the rate of flow through an identical package will be reduced to  $100 \times 1/310 = 0.3\%$  (see Eqn. 2) of the flow of water. A value of 0.3% represents a reduction of the order obtained in the experiments described and, therefore, a simple mechanical explanation is sufficient to account fully for the behaviour. The viscosity, or rigidity, of foam will vary with the agent used. Very little is known of this effect, although brewers have investigated it<sup>4</sup>.

#### More Practical Implications

##### PRODUCTION OF FOAM

In the experiments described foam was produced in the dyeing system by blowing a stream of air through the dye liquor below the textile package. These are obviously artificial conditions. In practical dyeing foam can be formed as a result of—

- (1) air being sucked in by leaking glands in circulating pumps,
- (2) dissolved air in the water being released as the temperature is raised,
- (3) carbon dioxide being released as the temperature is raised (carbon dioxide is present in considerable amounts in softened water originally of high temporary hardness),
- (4) heating with an open steam pipe (as the temperature approaches 100°C the steam does not condense rapidly),

- (5) mechanical action on or under the surface of the dye liquor, which can result in air being pulled into the liquid to produce foam,
- (6) air being trapped in the textile package (air is always present and can readily produce foam which, if stable foaming conditions are present, can be very difficult to remove, particularly with fibres having a hydrophobic surface).

#### EFFECT OF RESIDUAL SCOURING AGENT

Very little is known of the relation between the chemical constitution of an agent and its ability to produce foam. Not all surface-active agents are foam producing—in fact, the silicone antifoaming agents produce an appreciable lowering of the surface tension. It is thought<sup>5</sup> that the rate at which a molecule of the agent can migrate to the surface of the interface is the controlling factor. It is also known<sup>6</sup> that many agents which are foam-producing in themselves do not foam when in mixtures. For example, both alkyl sulphates and soaps are foaming agents, but in admixture produce little foaming. This kind of behaviour is clearly of interest to the practical dyer as it means that, quite apart from the problem of producing a clean scour, the nature of the scouring agent and auxiliary product used in dyeing can, if conditions which encourage foaming are encountered, make all the difference between a level and an unlevel dyeing. This difference results from the interaction of the two agents either to encourage or to discourage foaming.

#### EFFECT OF DYEING pH

Most of the present work on liquor flow was carried out at a pH of approx. 3, since under these conditions a clear picture of the influence of the various factors on the flow of dye liquor through wool emerged. At pH 7 where, as practical dyers are aware, flotation is common the picture became a little blurred. It is suspected that flotation in neutral-dyeing conditions, even in absence of an auxiliary product, is caused by the slight breakdown of the wool which is known to take place under these conditions, releasing soluble protein into the dyebath; soluble proteins stabilise foam<sup>7</sup>.

#### INFLUENCE OF THE CIRCULATING SYSTEM

Fig. 4 shows that the viscosity of the foam is rapidly reduced as the shearing force is increased. Therefore, there is less likelihood of difficulty being caused by foam formation in a machine with a powerful circulating system than in one with a poor system, provided that the powerful system does not produce a greater quantity of foam by the introduction of air. Here also lies one of the reasons for the superiority of the two-stick hank-dyeing machine over the single-stick machine. It is believed, however, that the information presented in this paper could lead to a more efficient use of a one-stick Hussong machine. Two changes from general practice are suggested—

- (1) addition of antifoam before the dye and dyeing assistants (on a one-stick machine the flow is very sensitive to foam formation),
- (2) a much greater use of downward rather than upward flow on the machine.

Upward flow should be given only to the minimum required for the prevention of stick marks. The reason for this suggestion is that on downward flow there is less possibility of failing to achieve penetration of the hanks in the machine by dye liquor, and, in fact, conditions are similar to those in a two-stick machine, provided that flotation is prevented by the correct addition of antifoaming agent.

#### Conclusions

The presence of foam in the dyebath can seriously affect the circulation of dye liquor through a textile package. This effect can be sufficiently large to become of comparable importance to uniformity of pH, migrational properties of dye, and rate of dyeing, and can, therefore, produce results completely contrary to those expected from a laboratory evaluation.

Many auxiliary products used to improve the level-dyeing properties of wool dyes encourage the production of foam in certain dyeing systems. In these cases, unless precautions are taken, what is gained by improving the dyeing properties is out-weighed by reduced circulation of dye liquor.

Antifoaming agents are of great value in nullifying the deleterious effects of foam in the dyeing system, but the time and place of addition must be carefully chosen if they are to be effective. It is important, where possible, to add the antifoaming agent to the dyeing system before the foam-producing agents.

Consideration of the mechanical properties of foam is sufficient to account for the results obtained.

\* \* \*

The authors wish to express their appreciation to the many practical dyers with whom useful discussions have been held which have helped considerably in the work described.

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(Received 10th February 1961)

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## CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

## Effect of Ultraviolet Radiation on the Dyeing Properties of Worsted Fabric

Exposure of wool to ultraviolet (u.v.) radiation increases the rate of absorption of acid dyes<sup>1</sup>. According to Haly<sup>2</sup> this effect is due to modification, but not destruction, of the fibre epicuticle. Work has been carried out to see if the effect could be applied to the printing of worsted fabrics. After starting the work we learned that knitted goods had already been printed on a semi-commercial scale<sup>3</sup>; our work benefited from exchanges of information with the patentees, to whom we are grateful.

A  $2 \times 2$  twill worsted fabric of weight approximately 8 oz/yd<sup>2</sup> was used. Samples were irradiated by means of a Philips 3 kW mercury-in-quartz lamp (HOK 3000W), placed 3 ft away from the fabric to prevent scorching. At this distance a semicircular reflector of aluminium produced a focused region 15 in. wide, and all irradiation times are referred to this region and the above conditions. Tests were made on 36 in.  $\times$  3 in. strips, which were dyed on a small-scale winch. All dyes used were of the level-dyeing acid and milling acid types, and were applied from a

standard bath of composition (wt./wt.)—sodium sulphate (anhyd.) 10%, sulphuric acid (conc.) 5%, Taninol WR 1%, dye 1%. The sample strips were irradiated in steps from an initial 15 s to a final 40 min duration, and an untreated area was left along one side as a control.

To measure contrast on the samples, spectral curves of the treated and control areas were plotted on a General Electric reflectance spectrophotometer. Reflectances were calculated as a single-figure approximation by the Selected Ordinate method<sup>4</sup>, and the degree of contrast given by each dye was expressed as the ratio of the reflectance of the control area ( $R_c$ ) to that of the irradiated area ( $R_i$ ).

Degree of contrast at constant dosage was greatly dependent upon the dye used. Thirty-three dyes were used, selection being mainly at random, although groups of dyes differing only slightly in composition were included. Table I shows the contrast values obtained after irradiating the cloth for 40 min.

TABLE I  
Contrast Values

Dye	Colour Index No.	Contrast
	C.I. Acid	
Erio Fast Cyanine S	Blue 45	3.9 R*
Novazol Acid Blue GL	Blue 121	3.7 R
Lissamine Blue BF	Blue 18	3.6 R
Erio Fast Green GS	Green 25	3.4 R
Erio Anthracene Blue R	Blue 47	3.3
Sella Fast Dark Blue B	Blue 60	3.1
Wool Fast Green B	Green 19	3.0
Bordeaux B	Red 17	2.9 R
Erioglaucine E	Blue 9	2.8 R
Coomassie Violet 2R	Violet 9	2.8
Lissamine Green SF	Green 5	2.7 R
Brilliant Wool Blue BLN	Blue 59	2.6
Naphthol Red B	Red 27	2.5 R
Naphthalene Red EA	Red 13	2.4 R
Erio Glaucine X	Blue 7	2.4
Fast Light Acid Green	Green 1	2.3
Polar Brilliant Blue GAW	see Blue 127	2.2
Lissamine Fast Red 3G	Red 5	2.1 R
Naphthalene Scarlet 4R	Red 18	1.9 R
Kiton Fast Red G	Red 1	1.8 R
Cloth Fast Blue S5R	Blue 113	1.8
Coomassie Red PG	Red 85	1.6
Amacid Fast Orange LW	Orange 31	1.6 R
Naphthalene Fast Orange 2G	Orange 10	1.5 R
Naphthalene Red J	Red 88	1.4
Metanil Yellow VS	Yellow 36	1.4
Naphthol Yellow SNA	Yellow 1	1.4 R
Coomassie Yellow R	Yellow 42	1.4
Acid Yellow T	Yellow 23	1.3 R
Naphthalene Orange G	Orange 7	1.3
Solochrome Flavine R	Yellow 8	1.3
Erio Fast Yellow RS	Yellow 25	1.3
Erio Yellow S	Yellow 27	1.3 R

\* R indicates that the irradiated zone took up less dye than the control zone at low dosages.

With approximately half of these dyes reversed contrast was evident at low u.v. dosages, i.e. the irradiated zone took up less dye than the control zone. At higher dosages contrast became normal. Reversed contrast changed to normal contrast after about 8-10 min exposure, except with Novazol Acid Blue GL, Erio Fast Green GS, and Naphthol Yellow S, where the reversal effect was small and normal contrast was observed after 5 min.

It is apparent from Table I that contrast is colour dependent. This probably follows from the method of measurement, which was intended to correspond to subjective judgment. If the contrast were measured in terms of the ratio of the dye concentration in irradiated wool to that in un-irradiated wool, the order would probably be different.

The degree of contrast can be increased, e.g. by adding Taninol WR or a similar inhibiting agent (cf. work on chlorinated wool<sup>5,6</sup>) to the dyebath. On the other hand, addition of a levelling agent (Neovadine AN) resulted in a very slight decrease in contrast.

Again, a pretreatment (alkaline chlorination and peroxide bleach) markedly increases contrast on subsequent irradiation<sup>7</sup>. Bleaching destroys the yellow colour produced by chlorination. Yellowing is also produced, but to a smaller extent, after u.v. irradiation, and can also be remedied by bleaching, which does not affect the differential dyeing properties.

No correlation was found between the degree of contrast and the molecular constitution of the dye. In work concerned with the contrast obtained when dyeing mixtures of chlorinated and unchlorinated wools<sup>5</sup>, it was shown that in a series of dyes [Naphthalene Red J (4'), Naphthalene Red EA (4',7), Naphthalene Scarlet 4R (4',5,7)] of the same basic structure, the contrast increased as the number of sulpho groups in the dye molecule increased (positions shown in parentheses). The same dyes, with the addition of two others having sulpho groups differently positioned [Naphthol Red B (2',4',7) and Bordeaux B (2,7)] were tested on irradiated samples. Table I shows that, although Naphthalene Red J occupies a similar low place in the scale of contrast, there is no obvious parallel with the results for chlorinated wool.

In general, u.v. irradiation enables cloth to absorb dyes at a greater rate than normal, and apparently also to retain a higher equilibrium concentration of dye. Similar effects on rate of dyeing are obtained by chlorination under acid conditions<sup>5,6</sup>, but two differences exist. Firstly, with u.v.-treated wool every dye tested gave some contrast, and secondly, work on chlorinated wool has not revealed a parallel to the reversal effect.

These observations suggested that u.v. irradiation might be used for printing on wool textiles. A set of samples (3 ft x 1 ft 6 in.) was therefore prepared and irradiated. From the results it was evident that the method held promise; fine detail was possible and a wide variety of designs could

be envisaged. Examples of the patterns obtainable are shown in Fig. 1. However, disadvantages were also apparent—

- (a) irradiation times were too long,
- (b) the process gave only tone-in-tone effects, which are not as desirable as the multicolour patterns obtained by conventional printing techniques,
- (c) it was impossible to produce a pure white without recourse to resist methods.

Though cloth printing by this process is not yet considered commercially practical, other uses may be found for u.v. irradiation in textile processing. Two possibilities are—

- (a) to replace chlorination, where this is used as a pretreatment for screen-printing or dyeing, and
- (b) to reduce the exhaustion time of the dyebath.

To check the efficiency with respect to (a), three pieces of cloth (irradiated, chlorinated, and control) were screen-printed\* with a simple block pattern in five colours. The intensity of the coloured areas was in each case greater in the treated samples than in the control, and differences were also observed between the irradiated and the chlorinated pieces. These differences were very slight and random. It was also noticed that the chlorinated cloth had yellowed considerably more than the irradiated one and had a much harsher handle. However, a hydrogen peroxide bleach before printing removes the yellow colour and softens the handle to some extent.

Because of the unpleasantness associated with the handling of chlorine, irradiation, with its inherent ease of control, could well replace chlorination in this field, provided always that cost and treatment times were comparable.

Pieces of irradiated cloth were dyed with each of four dyes at a concentration of 1%. Pieces of untreated cloth were then dyed at increasing dye concentrations till their colour intensities matched those of the irradiated samples. The concentrations (%) of dye required to achieve matching were—

Lissamine Green SF	2.6
Lissamine Blue BF	2.8
Erio Yellow S	2.2
Kiton Red G	1.7

Measurements were also made of the dyebath exhaustion times. Dyebaths containing irradiated fabrics were exhausted in about 30 min, whereas those containing untreated fabrics were exhausted only after 3-4 h.

Of the dyes used, four were selected for tests of washing and light fastness, on the basis of their Deutsche Normen ratings as given in the *Colour Index*. They were—

Lissamine Green SF	Poor wash fastness
Wool Fast Green B	Good wash fastness
Naphthol Yellow S	Poor light fastness
Erio Fast Green GS	Good light fastness

Two pieces of cloth, one a control, the other irradiated for 30 min, were dyed to the same

\* By Tennison Textiles Ltd, to whom thanks are due.

colour intensity with each of these dyes, and their fastness properties tested.

For the dye of poor washing fastness, there was no appreciable difference between treated cloth and control, but with that chosen as being good in this respect, fastness was reduced by one unit as a result of u.v. irradiation.

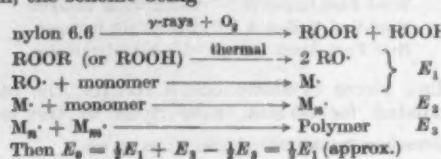
In light-exposure tests, both treated and untreated cloths showed differences after 45 h exposure, whatever the dye used. The colour of cloths dyed with Naphthol Yellow 8 had changed to fawn, but the irradiated cloth was considerably browner. The change in colour of the cloth dyed with Erio Fast Green GS was much less pronounced, the irradiated sample being very slightly darker.

The high cost of u.v. irradiation of wool is probably the main barrier to its use in the textile industry. If a lamp is replaced every 1000 hours (when loss of intensity is about 30%), the cost of irradiation (power cost and lamp depreciation only), is about 1s. 3d.(A) per yd of fabric, for material exposed for 30 min to a 3-kW lamp. Of this amount nearly 1s. 0d.(A) is set aside for depreciation. Thus, substantial reduction in processing cost requires a lamp at least as efficient as that used, and with a longer effective life.

#### Polymerisation of Methacrylic Acid in Irradiated Nylon 6.6 Fibres

Studies of the rate of the internal polymerisation of a vinyl monomer inside textile fibres, e.g. wool and nylon, are complicated by the rate of diffusion of the monomer within the fibre. Valentine<sup>1</sup>, who studied the rate of deposition of polyacrylonitrile in wool fibres, using the ferrous ion-peroxide catalytic system developed by Lipson and Speakman<sup>2</sup>, found that the amount of polymer deposited was initially dependent on the square root of the time of polymerisation. He concluded that diffusion of monomer into the fibre was the rate-controlling step, since the rate of such diffusion processes is generally dependent initially on the square root of the time, whereas that of radical polymerisations is usually directly proportional to time. For any valid study of the internal polymerisation, diffusion of monomer into the fibre must be completed first.

Roberts and Thomas<sup>3</sup> have recently published the results of an investigation into the grafting of polymethacrylic acid on to nylon 6.6 fibres, using nylon previously irradiated in air by  $\gamma$ -rays from a  $^{60}\text{Co}$  source. The fibres were placed in aqueous methacrylic acid solutions under vacuum, and the amounts of polymer formed both in the nylon and in the solution determined at various times. An activation energy ( $E_a$ ) of  $13 \pm 1$  kcal. mole<sup>-1</sup> was found, and was interpreted as being half the activation energy of formation of radicals from peroxide sites formed within the nylon by irradiation, the scheme being—



Further improvements may be effected by pretreating the wool with photosensitising reagents. Several absorbers, i.e. anthracene, casein, benzoyl-resorcinol and benzophenone, have been tested; only benzophenone showed any promise. The cloth was sprayed with a 0.2-M alcoholic solution of benzophenone, irradiated, and the reagent removed with hot alcohol before dyeing. This treatment allowed a reduction of irradiation time by a factor of approximately one-third. It is felt that possibilities in this direction are by no means exhausted.

G. L. STOTT

C.S.I.R.O. WOOL RESEARCH LABORATORIES  
DIVISION OF TEXTILE PHYSICS  
RYDE, N.S.W.  
AUSTRALIA

4th November 1960

- 1 Speakman, J. B., and McMahon, P. R., *New Zealand J. Sci. Technol.*, **20**, No. 5B, 248B (1939).
- 2 Haly, A. R., *Text. Research J.*, **28**, 182 (1958).
- 3 Harrison, H., & Co. (Finishers) Ltd. *BP* 811,702; *J.S.D.C.*, **75**, 330 (1959).
- 4 Hardy, A. C., and Staff of Massachusetts Institute of Technology, *Handbook of Colorimetry*.
- 5 Cross Dyeing of all-Wool Fabrics Containing Chlorinated Wool. *Wool Sci. Review*, No. 16, 15 (1956).
- 6 Barritt, J., and Elsworth, F. F., *J.S.D.C.*, **64**, 19 (1948).
- 7 Moore, D. R., and Harrison, H., and Co. (Finishers) Ltd. Private communication.

We have been studying the diffusion of methacrylic acid within nylon 6.6 fibres at various temperatures, and find an activation energy of 13 kcal. mole<sup>-1</sup> for the diffusion coefficient for the initial sorption. The amounts of monomer sorbed depend on  $(\text{time})^{\frac{1}{2}}$  up to about 80% of saturation. When the data of Roberts and Thomas for the amounts of polymer formed in the fibres are plotted against  $(\text{time})^{\frac{1}{2}}$  (Fig. 1), reasonably good straight lines are found. It is therefore clear that the

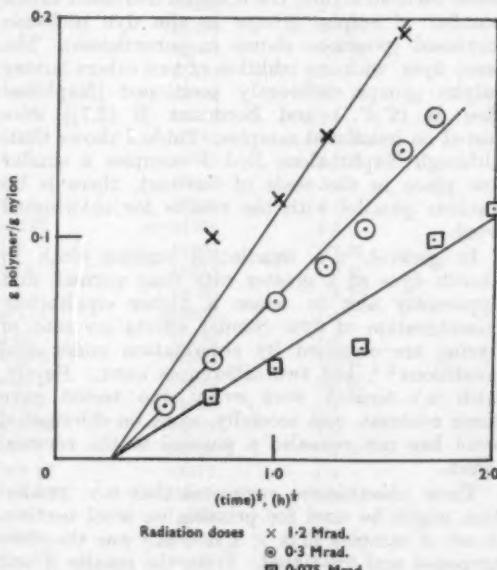


FIG. 1—Dependence of the amount of polymer deposited on the square root of time at 75°C

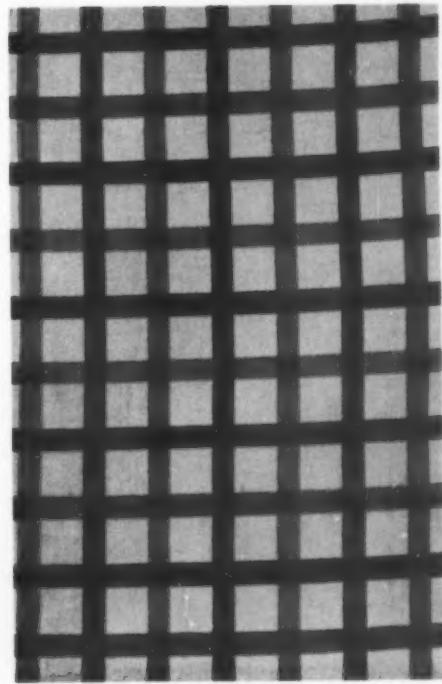
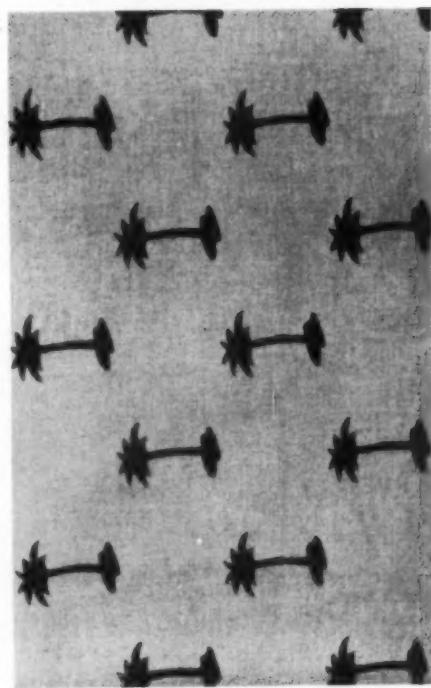
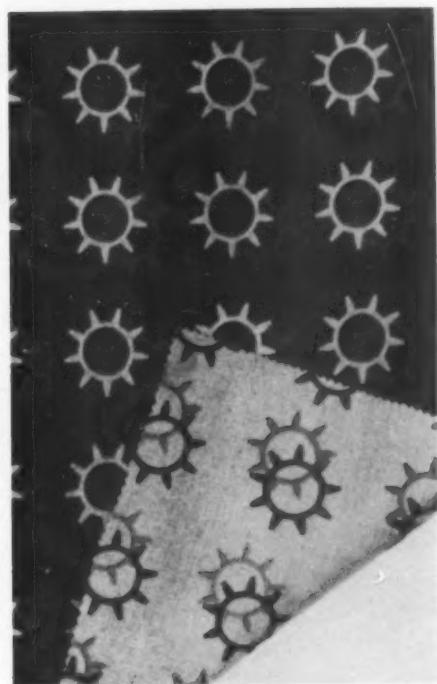
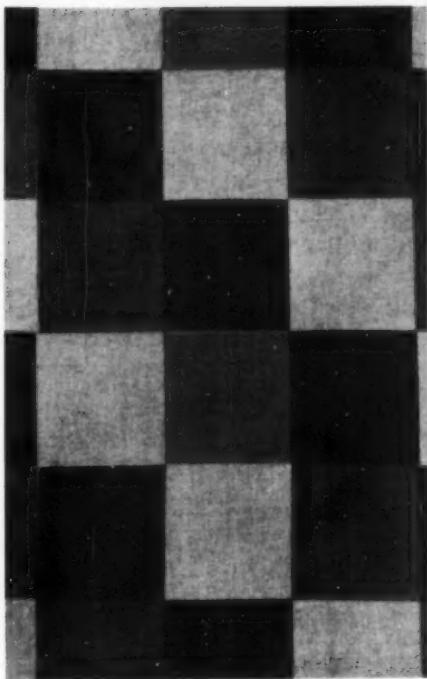


FIG. 1

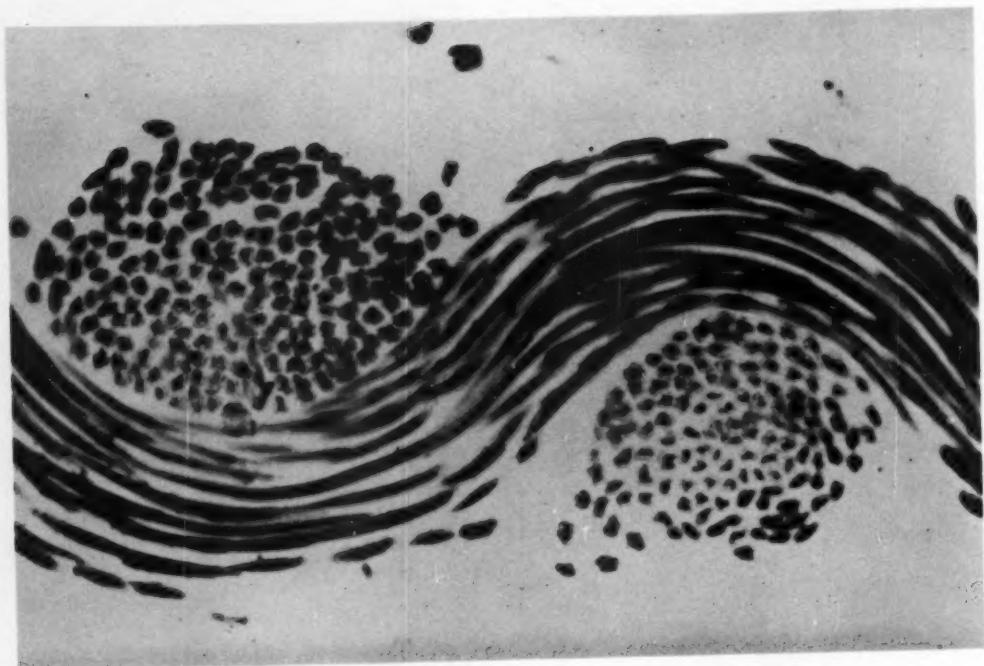


FIG. 1—( $\times 208$ )

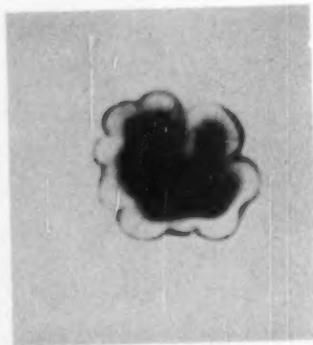


FIG. 2—( $\times 1640$ )

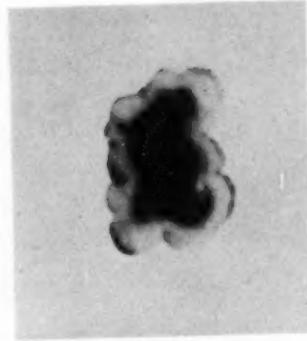


FIG. 3—( $\times 1640$ )

formation of polymethacrylic acid in their experiments was determined by the rate of diffusion of monomer into the fibres and not by the rate of radical formation from the peroxides. Fig. 1 shows that there was a short induction period; we have found this too.

It is questionable if such a scheme as that given can represent this polymerisation, since termination by mutual reaction of growing chains is an unfavourable process in a solid system, as suggested by Morawetz and Fadner<sup>4</sup> for the solid-state polymerisation of acrylamide. Since the amounts of polymer deposited are determined by the rate of diffusion of monomer, the rate of the internal polymerisation must be considerably greater than this, and therefore much greater than the rate of the corresponding solution polymerisation. This is consistent with a reaction scheme in

which mutual termination of growing chains plays at most only an unimportant rôle. Roberts and Thomas analysed the solution to determine by difference the amount of polymer in the fibres, but monomer is lost from the solution by simple sorption into the fibres, without it all necessarily being polymerised at any given time. Our results on the sorption and diffusion of methacrylic acid in nylon 6.6 fibres will be published elsewhere shortly.

A. R. MATHIESON

DEPARTMENT OF TEXTILE INDUSTRIES  
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LEEDS 2

3rd February 1961

<sup>1</sup> Valentine, *J. Textile Inst.*, **46**, T270 (1955).

<sup>2</sup> Lipson and Speakman, *J.S.D.C.*, **65**, 390 (1949).

<sup>3</sup> Roberts and Thomas, *J.S.D.C.*, **76**, 342 (1960).

<sup>4</sup> Morawetz and Fadner, *Makromol. Chem.*, **34**, 162 (1959).

### Location of Resin Finish in Viscose Rayon Fabrics

A recent paper by Park<sup>1</sup> provides evidence that largely vindicates the microscopical staining tests<sup>2</sup> for the location of amino-resin finishes in cellulosic fabrics. Park's autoradiographic method shows that, in fabrics that have been normally impregnated with resin, the greatest resin concentration is in fibres near the fabric surfaces, and our experience of staining these fibres agrees with this view. Fig. 1 shows a stained section of a typical spun-rayon fabric that had received a crease-resistant finish with urea-formaldehyde resin; the dye is Solway Blue BN (C.I. Acid Blue 45). The staining shows that all the fibres contain some resin, but the fibres nearest to the two surfaces of the fabric are the most deeply stained, and presumably the more heavily impregnated with resin as a result of migration of the precondensate during the preliminary drying. The similarity between this stained specimen and the autoradiograph that is Fig. 4 in Park's paper is striking.

A more detailed inspection of the original preparation shows evidence of deeper staining towards the centres of those individual fibres that, by virtue of their particular position in the twisted yarns, lie normal to the plane of cutting. (Most of the individual fibres are, of course, sectioned at various degrees of obliqueness.) This feature has been investigated more closely on separated fibres and there is no doubt that the staining is more intense in the central region, with a band of lower concentration next to the fibre surface. This is illustrated in Fig. 2, which shows a transverse section of a single fibre (Fibro) of 1.5 denier taken from the resin-finished fabric and stained with Solway Blue BN. This experiment recalls the observations of Landells, Mhatre, and Narasimhan<sup>3</sup>, and of Kramer and Graeser<sup>4</sup>. Fig. 3 shows a section of a single fibre from the unfinished fabric, stained by a method based on that of Hermans<sup>5</sup> so as to reveal the core region.

From the correspondence between the stained regions in the two figures there can be no doubt that the apparent preferential uptake of stain in Fig. 2 is confined to the core structure, and that the difference in uptake between skin and core is

considerable. Joshi and Preston<sup>6</sup> have shown that in conventionally dyed fibres the core contains more dye than the skin; in view of this observation it must be emphasised that the appearance in Fig. 2 is not due to substantivity of the dye to the cellulose of the core, since untreated fibres are not dyed by the acid dye used for resin staining. Hence it is safe to assume that the staining difference shown in Fig. 2 is due entirely to a different content of resin in skin and core, despite the fact that the components of the resin must have diffused radially through the skin in order to occupy the presumably more favourable sites for resin formation available in the core.

The acceptance of this observation necessitates qualification of Park's statements concerning the uniformity of resin distribution within single fibres, and indicates that the skin-core structure plays an important part in the resin impregnation of viscose rayon.

J. E. FORD

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SHIRLEY INSTITUTE  
DIDSBURY  
MANCHESTER, 20

23rd February 1961

<sup>1</sup> Park, *J.S.D.C.*, **76**, 624 (1960).

<sup>2</sup> Marsh, *Text. Manuf.*, **85**, 35 (1959).

<sup>3</sup> Landells, Mhatre, and Narasimhan, *J. Textile Inst.*, **39**, T148 (1948).

<sup>4</sup> Kramer and Graeser, *Melliand Textilber.*, **33**, 226 (1952).

<sup>5</sup> Hermans, *Text. Research J.*, **18**, 9 (1948).

<sup>6</sup> Joshi and Preston, *ibid.*, **24**, 971 (1954).

The above communication by J. E. Ford draws attention to the evidence of staining tests that resin-finished viscose filaments contain a much higher resin concentration in the filament core than in the skin. In general, owing to the similarity of the magnitude of the autoradiographic resolution and the skin thickness of textile yarn, our published autoradiographic studies<sup>1</sup> do not provide evidence about this. Recently, however, we have started an autoradiographic study of the distribution of the resin finish in several tyre-cord yarns with different skin:core ratios. One of these yarns has already

shown a greater resin concentration in the filament centres than in the outer regions. This is only a preliminary observation and it is hoped to publish a more detailed communication when the work has been completed.

G. S. PARK

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14th March 1961

<sup>1</sup> Park, J.S.D.C., 76, 624 (1960).

### Man-made Fibres

Readers of the *Journal* will recollect the correspondence some time ago about the word "man-made", and I remain most shocked at having to use this monstrous hyphenated, unintelligent, unintelligible, unscientific, incorrect, meaningless, inadequate, nonsensical, preposterous, and absurd word. I remain unaware that silk is referred to as a "caterpillar-made" fibre in normal reference nor is wool normally called "sheep-made", although the terms would be correct. If the hyphenated absurdity "man-made" meant anything at all, a Venusian or Martian would gain the impression that human beings (Earthians?), in some strange way, could produce through an appropriate spinneret a glandular, say salivary, liquid which turned into a

fibre on contact with air. "Man-made" is not even pronounceable by those not knowing the English tongue and, therefore, has not the international sound of, e.g., "rayon", even when the latter is spelt "reyon", as on the Continent. The complete absurdity of the word is appropriately shown if I write that I am sitting on a "man-made" chair next to a "man-made" desk writing on "man-made" paper using a "man-made" pen containing "man-made" ink, and so on, *ad nauseam*. How perfectly preposterous and yet how true!

German and Swiss chemists sensibly use the word "Chemiefaser", i.e. they say rightly that they are "chemical fibres". Hence I submitted at the time that we should use internationally a generic word such as "Chemifil"; for the names of specific fibres one could consider "filamide" and "filester", or "amidofil" and "esterfil", and so on.

I feel bound to add that some of us are not happy that most countries now refer to the fibres under discussion as "synthetic" since, obviously, they are not the result of a synthesis of any product as indigo was. Alternatively we require another word for a product made as the result of a synthesis.

H. A. BRASSARD

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LONDON N6  
27th February 1961

## Notes

### Oil and Colour Chemists' Association (London Section) Thirteenth Technical Exhibition 6th-9th March 1961

For those engaged in the industries served by the Oil and Colour Chemists' Association (broadly speaking, those of surface coating for protection or decoration) the Annual Exhibition is a compulsive event; two things conspire to make this so. First, this is an unrivalled opportunity for discussion among the technicians from both sides of this somewhat specialised fence, suppliers and consumers, and the very nature of the exhibition ensures a concentration of interest. Second, and scarcely less important, the restricted size of the exhibition makes this the one such event in the average year that offers the visitor a reasonable chance of seeing everything within a full day's saunter. By 1957 the OCCA Exhibition had reached the limit of accommodation offered by either the New or the Old Hall of the Royal Horticultural Society, viz. some 80 stands, but this year, for the first time, the exhibition occupied both halls. The 100 stands thus housed represent almost the limit of capacity of the combined halls; they represent also the limit to which the exhibition can be allowed to expand without losing its manageable and intimate character, and the organisers should consider this carefully in their future planning.

The terms of reference of the exhibition are that "Exhibits will relate to new products, new

knowledge relating to existing products and their use, and, in certain cases, existing knowledge which is not generally available to the consuming industries." This has provided a splendid stimulus to the exhibitors to make their contributions technically meaningful and, therefore, worthwhile to the visitor in search of something more than a hospitable gin and tonic.

The surface coating and printing industries have in recent years undergone a deep-seated revolution — and one that is the more patent in view of the still heavy use of traditional materials. For every component in a coating composition there is a bewildering array of alternative materials, offering the possibility of meeting virtually any environmental demand. Binder materials of every type were exhibited by 29 firms, and there was significant emphasis on the current interest in coating systems that are water-dilutable or have water as the basic medium. Polyurethanes of improved light fastness were also much in evidence. Emulsion-paint technology was a feature on five stands.

Pigments, dyes, and extenders provided the basis for interesting features on 20 stands—daylight fluorescents of improved versatility and light-fastness (Swada, Cornelius), additions to the phthalocyanine range (Geigy, ICI), transparent brown cadmium pigments and non-toxic colorants

for printed effects on PVC (Matthey), a titanium-nickel yellow (Laporte), organic-solvent-soluble dyes for lacquers, enamels, and inks (Ciba Clayton), anti-fouling pigments (Rex Campbell), Hansa Yellow pigments of improved fastness and resistance to bleeding (Hoechst), chrome, Permanent Red colorants of better light fastness (ICI), and a violet colorant for the production of clean carbon papers (ICI). The exhibit of the Cornbrook Chemical Company was built around all types of red pigment, whilst that of British Titan Products showed how their titanias have been improved to take advantage of modern developments in milling equipment.

A wide range of solvents was displayed by nine major companies in this field, and six others stoutly defended the position of fats, oils, and their derivatives, those stalwarts of tradition.

A further 20 stands showed an immense range of plant for mixing, grinding, dispersing, emulsifying, filtering, proportioning and metering, distilling, handling, filling, dissolving, and heating. Here the emphasis was upon speed, efficiency, and flexibility. Manufacturers of electric drills of domestic dimensions now offer attachments to convert them into stirrers and mixers; it was therefore amusing to note that one plant manufacturer offered mixers provided with drill-type pistol grips.

#### Council Meeting—1st March 1961

Among the matters discussed at the meeting of Council on 1st March were—

**HONORARY MEMBERSHIP**—It was unanimously agreed that Mr. John Boulton, Immediate Past President of the Society, be made an Honorary Member of the Society. Mr. Boulton is the present President of the International Federation of Associations of Textile Chemists and Colourists. (The Diploma of Honorary Membership was presented to Mr. Boulton at the Annual Dinner, an account of which will appear in the July *Journal*.)

**ANNUAL GENERAL MEETING AND ANNUAL DINNER**—It was agreed that the 1962 Annual General Meeting and Annual Dinner be held in London. As the I.F.A.T.C.C. Congress will be held in Holland on 25th–27th April 1962, it was agreed that the date of the Society's A.G.M. and Dinner be brought forward. They will take place on 30th March 1962 in the Great Room, Grosvenor House, London.

**MEMBERSHIP**—46 Ordinary Members and 21 Junior Members were elected.

#### Acknowledgments in Papers

Publications Committee views with concern the growing practice of including, at the end of proceedings and communications, a long list of acknowledgments. The Committee feels that in the majority of cases such acknowledgment would be more appropriately made by the author in the form of a private letter, and that acknowledgment in the *Journal* should be confined to those who have made a major contribution to the work.

Apart from these broad and typical fields there was much evidence of the growth in the number of ancillary products designed to impart special properties to both coating compositions and finished coatings—slip agents, anticorrosion compounds, thickeners, stabilisers, driers, filter aids, grinding media and aids, antiskinning agents, fungicides, ultraviolet absorbers, fire-retardants, flattening agents, and plasticisers were but a few of these.

The more academic aspects of the field were also well cared for. Three research bodies had impressive displays, seven companies exhibited a wide variety of instruments for testing and control, and six other stands were devoted to publications (periodicals and textbooks), including, of course, that of OCCA itself.

Only the broadest survey of this compact and admirable exhibition has been possible, but it should serve to emphasise the immense value it has for the technologist. Further expansion in space is not desirable but an increase in the time allotted would be; in past years the exhibition has been open for three days, and the extension to four days made this year was thus a step in the right direction.

J. W. DUARTE

#### Waverley Gold Medal Essay Competition 1961

Research is sponsoring this competition for the ninth successive year. The competition is designed to encourage scientists and engineers to express their views and to translate their work into an essay (of about 3000 words) that will be readily understood by other scientists, directors of industrial firms, and others interested in science and technology. The first prize is £100 plus the Waverley Gold Medal, and the second prize £50; in addition a special prize of £50 will be awarded for the best entry from a competitor under the age of 30 on the last date of entry, 31st July 1961. Entry forms are available from The Editor, *Research*, 88 Kingsway, London W.C.2.

#### Spectroscopic Studies using Time Resolution Techniques

A one-day meeting on this subject, organised by The Institute of Physics and The Physical Society, will be held in Manchester on 16th June 1961. Detailed programmes and application forms are available from the Institute and Society, 47 Belgrave Square, London S.W.1.

#### Polymer Characterisation and Analysis

A special short course on Methods of Polymer Characterisation and Analysis will be held at the Bradford Institute of Technology on 23rd and 24th June 1961. Lectures will cover determination of molecular weights, molecular weight distributions, analysis of plastics and polymers (including i.r. spectroscopy), characterisation of block and graft copolymers, and determination of glass-transition temperatures. The fee for the course is £2 5s. 0d. Application forms are obtainable from, and should be returned to, The Registrar, Institute of Technology, Bradford 7.

### Electron Microscopy

The Electron Microscopy Group of The Institute of Physics and The Physical Society will hold its Annual Conference on 10th-14th July 1961 at the University of Nottingham. The Conference will be concerned mainly with the physics of the electron microscope and its applications. Application forms are available from the Administration Assistant, 47 Belgrave Square, London S.W.1.

### Federation of Societies for Paint Technology

The 39th Annual Meeting of the Federation and the 26th Paint Industries' Show will be held at the Shoreham Hotel, Washington, D.C., on 2nd-4th November 1961. Details may be obtained from the Federation, 121 South Broad Street, Philadelphia 7, Pa., U.S.A.

### Mrs. Mary Ard

The death recently occurred at the age of 82 of Mrs. Mary Ard, who was the last surviving child of Johann Peter Griess, F.R.S., a pioneer of chemistry whose work in London and Burton-upon-Trent earned him an honoured place in the history of

science. His discovery of diazo compounds and many dyes derived from them was of profound importance in laying the foundations of the modern organic chemical industry.

Mrs. Ard had lived in British Columbia for many years. To commemorate the centenary in 1958 of Griess's greatest discovery, Mrs. Ard presented to Imperial Chemical Industries Limited, Dyestuffs Division, many papers of historical interest formerly belonging to her father.

### Death of Member

We regret to report the death of Mr. F. Crompton.

### Meetings of Council and Committees

#### April

Publications—18th  
 Light Fastness Subcommittee—18th  
 Identification of Dyes—21st  
 Finance and General Purposes—21st  
*Colour Index* Editorial Board—24th  
*Review of Textile Progress*—27th  
 Annual General Meeting—28th  
 Annual Meeting of Chairmen and Secretaries of Sections—28th

## New Books and Publications

### Silicones

Edited by S. Fordham. Pp. xi + 252 + 8 plates. London: George Newnes Ltd. 1960. Price, 36s. Od.

This book will be welcomed by those wishing to keep abreast of the rapid advance made in the last 25 years in a fascinating field of science and technology.

The book is aimed at two types of reader. The scientist working in the silicone field will find the first part "Organosilicon Chemistry" of particular interest. This is subdivided into three chapters. The first, a brief historical introduction, is followed by a very comprehensive chapter on the chemistry of organosilicon compounds. Different methods of synthesising organosilanes are described, as well as numerous reactions of chlorosilanes, not least the reaction with water to form silanols and siloxanes (silicones). The second chapter closes with a brief, but useful, analytical section. The third chapter is devoted to the physical properties of silicones, and deals with bond strength, structure, and many other physico-chemical properties. These first three chapters occupy 106 pages, and at the end of each chapter there are numerous references to the literature and patents.

The second part, "Industrial Manufacture and Application" (139 pp.) should appeal to the general reader, as it presents an eminently readable account of the many existing and potential applications of silicones. It is subdivided into nine chapters. The first deals with the commercial aspects of silicones, including world production and markets, and future developments and trends; the second discusses the manufacture of silicones, including the manufacture, analysis, and distillation of

chlorosilanes, and the manufacture of the various forms of silicone polymers, namely fluids, resins, and gums. The next three chapters are devoted to a description of the properties and the many end-uses of silicone fluids, lubricants, rubbers, and resins, respectively. The last four chapters deal with electrical properties and applications of silicones, silicone masonry water repellents, textile applications, and miscellaneous uses, including applications to leather and paper, and uses in polishes and antifoams. A six-page subject index brings the book to a close.

It is inevitable to compare a new book on silicones with earlier specialised texts. Whilst E. G. Rochow's "An Introduction to the Chemistry of the Silicones" remains the standard reference book for the chemist working in this field, Dr. Fordham's new book has the advantage over R. R. McGregor's "Silicones and Their Uses" of being considerably more up-to-date. Even so, references in the book under review do not extend beyond 1958, an indication of the time required to get a collection of articles transformed and published in the form of a book.

It is not easy to fault Dr. Fordham's and his colleagues' efforts; one shortcoming is the repetition to which Dr. Fordham himself draws attention, but excuses on the grounds that it has cut down the amount of cross-referencing. Be that as it may, it is rather irritating to read several times the same statement concerning, e.g., the composition of silicone rubbers or the poor lubricating properties of silicone oils.

Another disappointment is the allotment of a relatively short space (only six pages) to the chapter on the treatment of textiles with silicones.

Although the review by Dr. J. A. C. Watt is commendable, it certainly presents only a bird's-eye view. For example, the joint use of crease-resist resins and silicones is dismissed in one sentence, and the use of silicones as softening agents in textile operations is not even mentioned.

With the exception of certain minor details, this book is lucidly written and well set out, and it is clearly printed on good-quality paper. It is a worth-while purchase for library or laboratory.

G. W. MADARAS

### Biological Degradation of Cellulose

By J. A. Gascoigne and M. M. Gascoigne. Pp. vi + 264. London: Butterworths & Co. (Publishers) Ltd. 1960. Price, 42s. 0d.

Readers whose interest in this subject has been stimulated by Dr. Gascoigne's paper "The Biological Chemistry of Cellulose", published in the February 1961 issue of the *Journal*, can be recommended to consult this book. Its aim is to summarise the enzymological background to the effects of cellulose decomposition by all living agencies in the textile, wood, paper, and fermentation industries, and in animal digestion.

The final chapter, which deals with the industrial significance of cellulases, is the one likely to be of most direct interest to the Society's members. Like all the other chapters it is very thoroughly documented, 331 references to the literature being included. Consideration is first given to the energy changes occurring during the complex sequence of chemical reactions which constitute breakdown of cellulose, and this is followed by sections on the degradation of cellulosic textiles, biological decay of wood, chemistry of wood decay, cellulases in the paper industry, prevention of enzyme action, cellulases in animal digestion, saccharification of wastes, and cereal enzymes. It is interesting to note that few universally accepted test methods for the evaluation of preservatives (rotproofing agents) exist.

The seven other chapters deal with the basic chemistry and physics of cellulose (174 refs.), enzymes involved in reactions of  $\beta$ -linked glycosides (115), sources of cellulases (120), purification of cellulases (162), properties of cellulolytic enzymes (92), mode of action of cellulolytic enzymes (76), and hemicellulases (43).

This book is well-written and contains a very extensive bibliography, which together make it an extremely useful addition to the literature.

M.T.

### Die Chemie der Applikation von Komplexfarbstoffen

By H. Gerstner. Pp. 202 + 21 fig. and 26 tables in the text. Berlin: Akademie-Verlag. 1959. Price (paperbacked), DM 25.00.

Since Dr. Gerstner was unable to include mordant dyeing in his earlier book, *Das Verhalten der direktzichenden Farbstoffe gegen tierische Fasern, Cellulose- und Kunstspinnfasern* (see J.S.D.C., 74, 590 (1958)), he has now written a separate volume. Its contents are outlined by the nine chapter headings—the history of Turkey Red dyeing,

mordants, alizarins and formation of their lakes, natural dyes as complexing agents, complex formation by dyes on the fibre, dyeing of protein and polyamide fibres with 1:1 metal-complex dyes, preparation and application of 1:2 metal-complex dyes, complex formation with polyacrylonitrile fibres, and other types of metal-complex dyes and their use. The book is limited to applications in the textile field, and uses on other fibrous substrates, such as leather, and as pigments are not dealt with.

Unfortunately, the present book exhibits the same faults as the earlier one. Thus, whilst the author has collected a useful list of more than 300 references, ranging from 1776 to 1958, the abstracts of these are presented in so undigested a form, practically without comment, as to make the volume very tedious. The reader is faced with a mass of facts, some of which may indeed be new to him, but his understanding is not likely to be increased.

It would have been better if Dr. Gerstner had begun with an exposition of current ideas on metal-complex formation and if the subsequent chapters had then been based on the application of these principles. As it is, many terms are introduced without definition—"formation constant" first appears on p. 91, "coordination number" on p. 117, and "penetration complex" on p. 181.

Overall the treatment is uneven. Some work is quoted at length (see pp. 34-39, 77-84, 129-133, 133-138, 150-152, 164-165, 167-169, 171-172, 190-193), yet other papers are dismissed very briefly, notably Schwarzenbach's researches, which rate only 10 lines. The preparation of a calcium lake of alizarin is described in detail, and the quantities used by Race, Rowe, and Speakman in their admittedly important work are given in full. Yet no complete recipe is given for the Metachrome process.

One important aspect not considered is the colour gamut obtainable by each of the various dyeing processes covered. Another is the relative importance of these processes in the different branches of the present-day textile industry. Little attention is paid to quantitative fastness data and to the problems of the dyestuffs manufacturer. The usefulness of the *Colour Index* is ignored. The chapter on natural dyes is superficial, maclurin, the catechins, and kermesic and carminic acids not being mentioned.

H. E. NURSTEN

### Untersuchungen zur Rationellen Durchfärbbarkeit von Bastfasergarnen (Forschungsberichte des Landes Nordrhein-Westfalen Nr. 761)

By I. Lambrinou-Geurten. Pp. 53. Cologne: Westdeutscher Verlag. 1959.

This research report, from the Technological Institute for Bast Fibre Industry, Bielefeld, is concerned with a detailed examination of the effect of various factors involved in the vat dyeing of linen yarns. The work was undertaken with a view to ascertaining the best procedure to give, on the one hand, good to completely level dye penetration with, on the other, minimum reduction of yarn strength and of the DP of the cellulose.

Three linen yarns, 35 lea (48 tex) line, 20 lea (84 tex) tow and 12 lea (140 tex) tow, previously bleached to 1/2-white on cross-wound spool, were vat dyed in three different ways, namely on cross-wound spool, in pack, and in hank. Three colours (blue, green, and salmon) in two depths (pastel and medium) were dyed in each case using Indanthren dyes by three different methods, namely normal, temperature stepwise, and the pigment process. Anthrasol dyes were also used in the production of the same colours. Full details of the processes and recipes are provided.

The dyed yarns were then examined subjectively by a panel of experts in order to classify them into three groups—(a) thoroughly, and (b) partially penetrated, and (c) surface dyed only. Results of tests of tensile strength, breaking length, extension at break, and of cellulose DP were compared with those of similar tests on undyed controls. Side issues, such as the effect of alkali concentration and of wetting agents on yarn characteristics, were also studied.

It is concluded that cross-wound spool dyeing, using Indanthren dyes, preferably, but not necessarily, by the pigment process, can give good to complete penetration and good levelness, irrespective of yarn lea and whether the fibres are in the form of line or tow, with little or no adverse effect on physical and chemical yarn properties. This method is also safest where higher alkali concentrations must be used. Hank dyeing, on the other hand, gives the poorest penetration, coupled with moderate to marked tendency to reduce yarn strength and cellulose DP. In this process wetting agents also can have a degrading effect, with little or no beneficial effect on penetration, although they do tend to give greater depths. The pack-dyeing method appears to give intermediate results for most factors. As might be expected, Anthrasol dyes are the safest to use by all three methods, but have little advantage over Indanthrens in the cross-wound spool process.

This document is well written, clearly printed, and its conclusions appear to be well authenticated. It should prove of interest to technologists working in this field, although it is probable that the information presented is not entirely new.

H. L. PARSONS

#### Standardization Activities in the United States—A Descriptive Directory

By Sherman F. Booth. Pp. iv + 210. Washington: U.S. Department of Commerce. 1960. Price, \$1.75.

This volume provides a descriptive inventory of the work of some 350 American organisations taking part in standardisation activities. Several of these are active in fields of interest to the Society. These include the American Association of Textile Chemists and Colorists, Color Association of the United States, Federation of Paint and Varnish Production Clubs (now the Federation of Societies for Paint Technology), Intersociety Color Council, National Association of Finishers of Textile Fabrics, National Paint, Varnish and Lacquer

Association, National Printing Ink Research Institute, and the Optical Society of America. A brief description is given of the particular interests and activities of each organisation.

The great extent of standardisation activities is revealed by a quick survey of the titles of the relevant bodies, not all of which are unambiguous, e.g. the Drop Forging Association (a released prisoners' aid society?) and the Elastic Fabric Manufacturers Institute (do the members work at full stretch?). One wonders, too, if membership of the American Society of Sanitary Engineering is restricted to specialists, and if the American Hot Dip Galvanizers Association excludes people with a proclivity towards sea-bathing in January.

A very useful index makes it possible to locate rapidly the bodies responsible for standardisation in any specific field.

M.T.

#### Britain's Scientific and Technological Manpower

By G. L. Payne. Pp. xiii + 466. Stanford: Stanford University Press, and London: Oxford University Press. 1960. Price, 45s. 0d.

This work is a very detailed study of the sources of our scientific and technological manpower and the use to which we put them. It is valuable not only on this account, but also because of the running comparison that the author makes between British and American practices in education and industry.

The work opens by surveying the economic position of the country and how it uses scientists and engineers among its manufacturing industries, making the case that it really is necessary for us to be in advance of the rest of the world in cultivating and deploying our skill since we, more than any other country commensurate in size and population, are so dependent upon exports to pay for our food and maintain our standard of living.

Everyone associated with the drive to promote science and technology in our universities and to break down some of the barriers which still exist in the older industries, and particularly in engineering, to the employment of university graduates, will find facts and figures to assist them in presenting individual cases to authority. We are all aware that very much activity is being encouraged not only in traditional universities and colleges of technology but also by the establishment of new centres of learning; nevertheless, there is nothing that leads us to believe that within the next ten or twenty years the number of graduates in Britain as a percentage of the population is likely to rise much from its present lowly place when compared with the percentage in the U.S.A. and in Western Europe.

The book covers in notable detail the use of scientists in research and development, and particularly in the Research Associations. The author, George Louis Payne, prepared the work at the request of the U.S. President's Committee on Scientists and Engineers. If this is representative of the approach of all the President's advisory committees, then we must take great heart, since he is in possession of a very accurate picture of the strengths and failings of our country.



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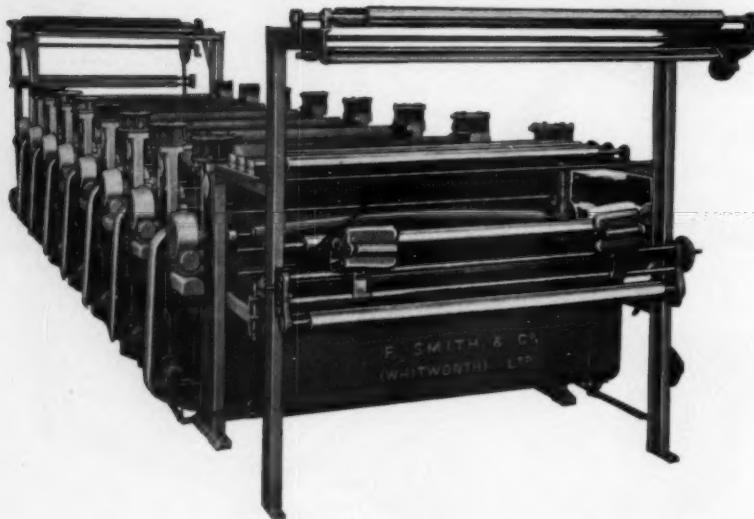
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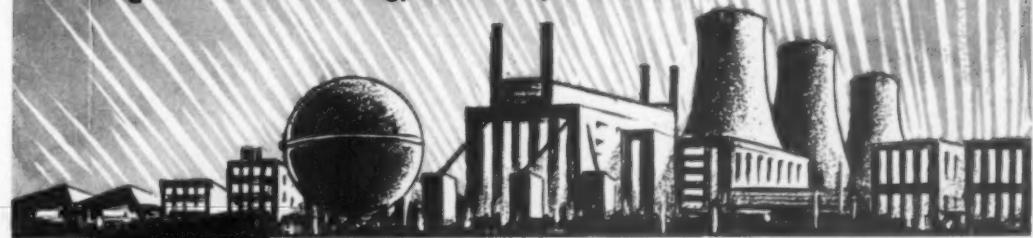
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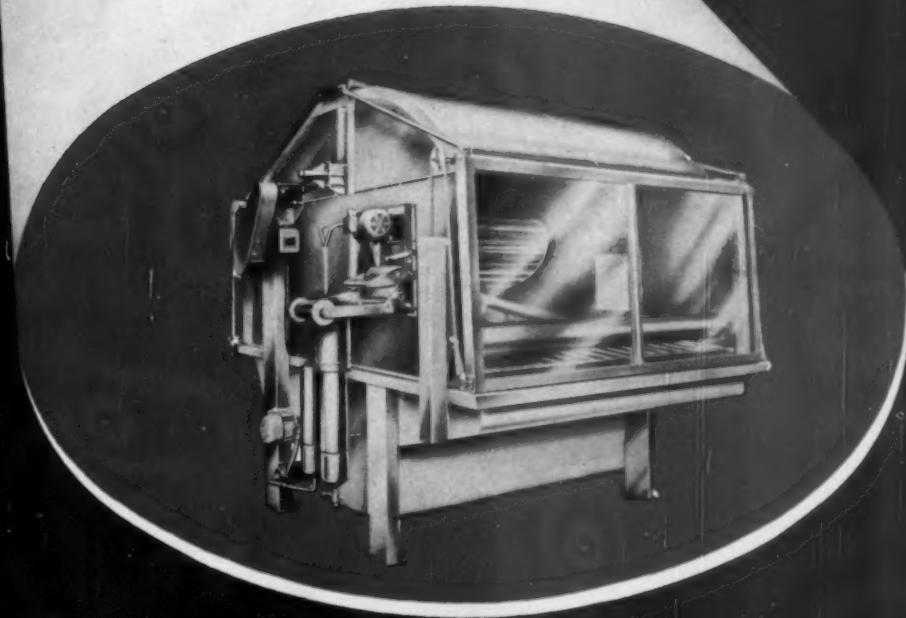
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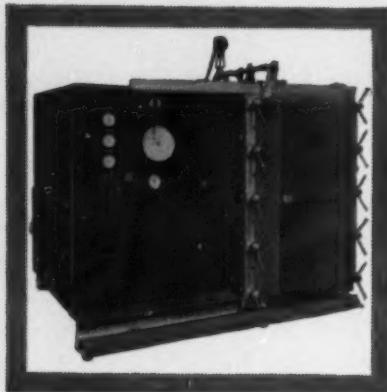
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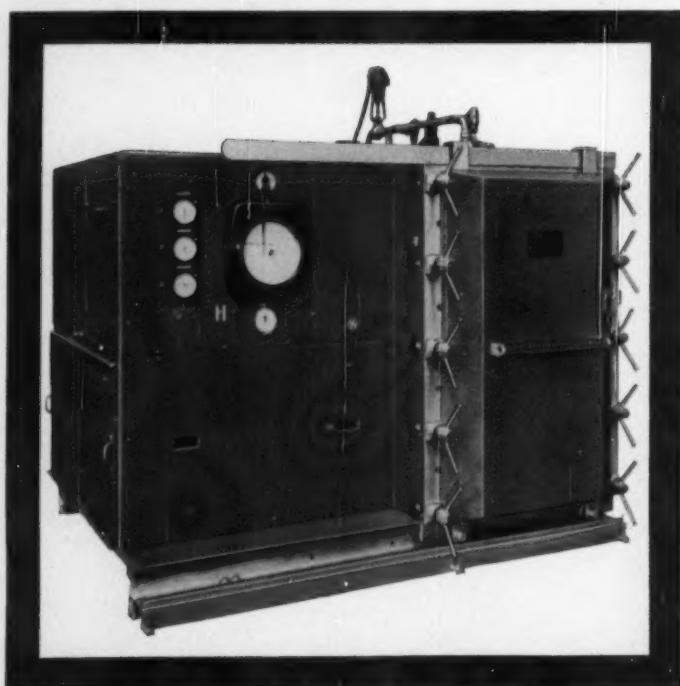
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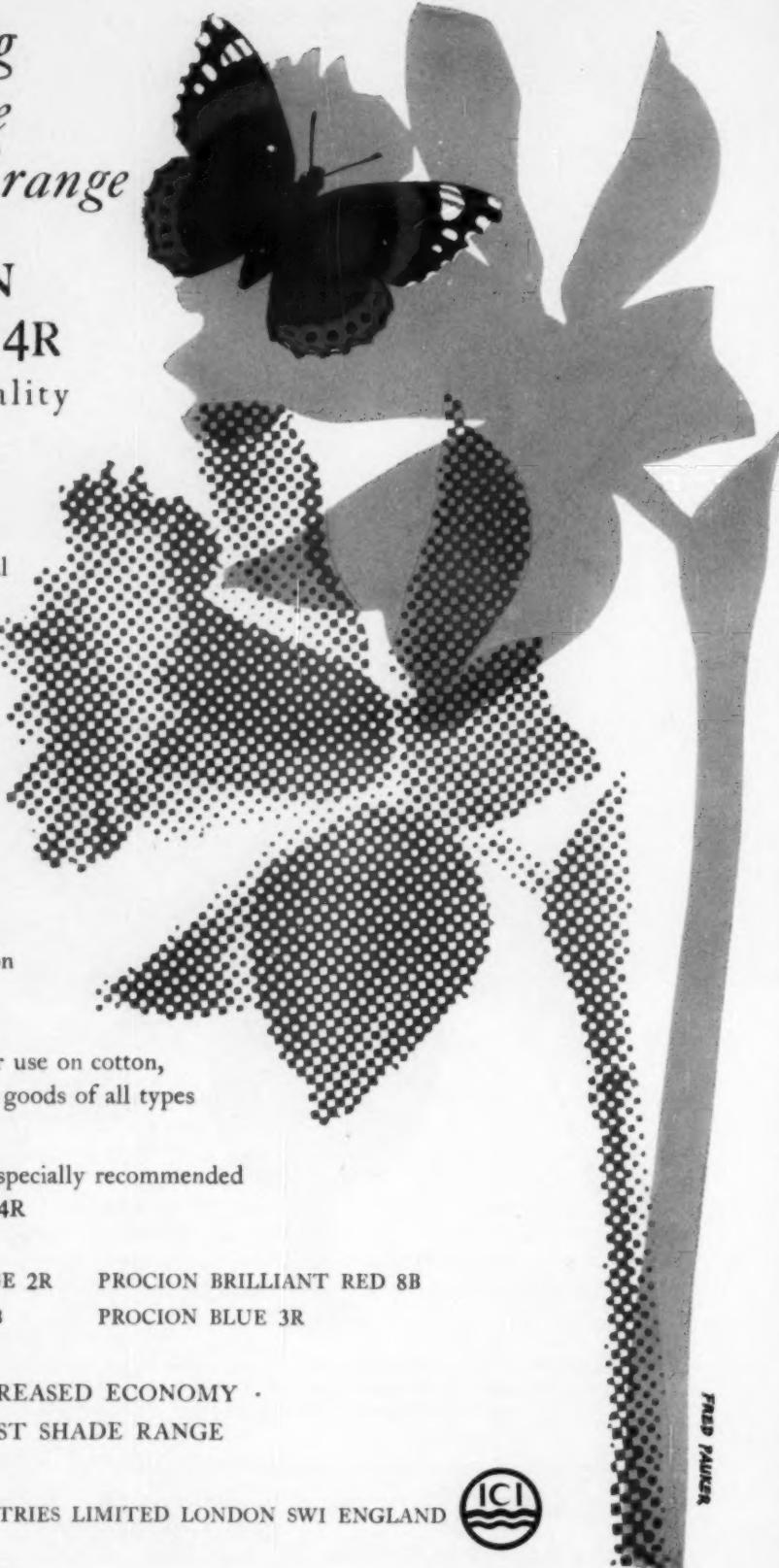
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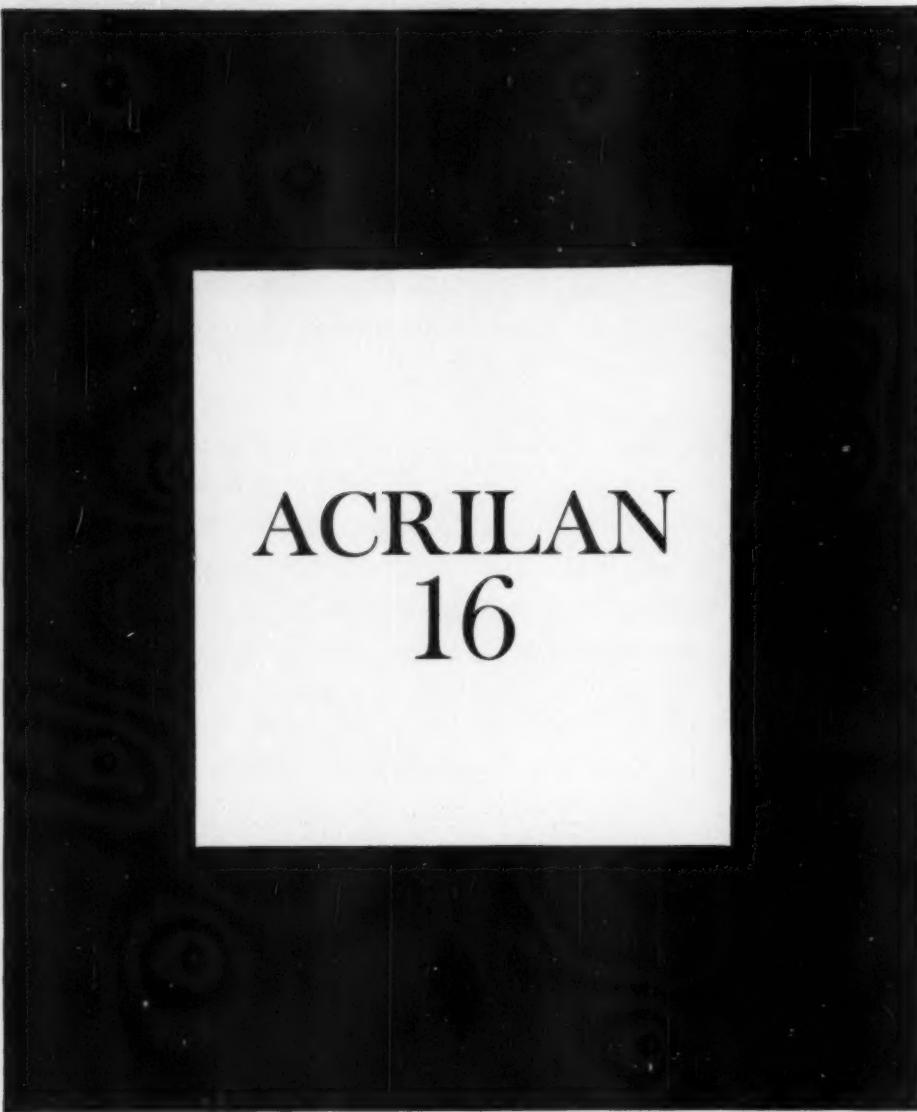
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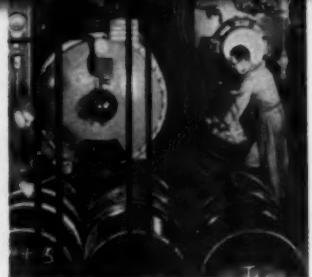


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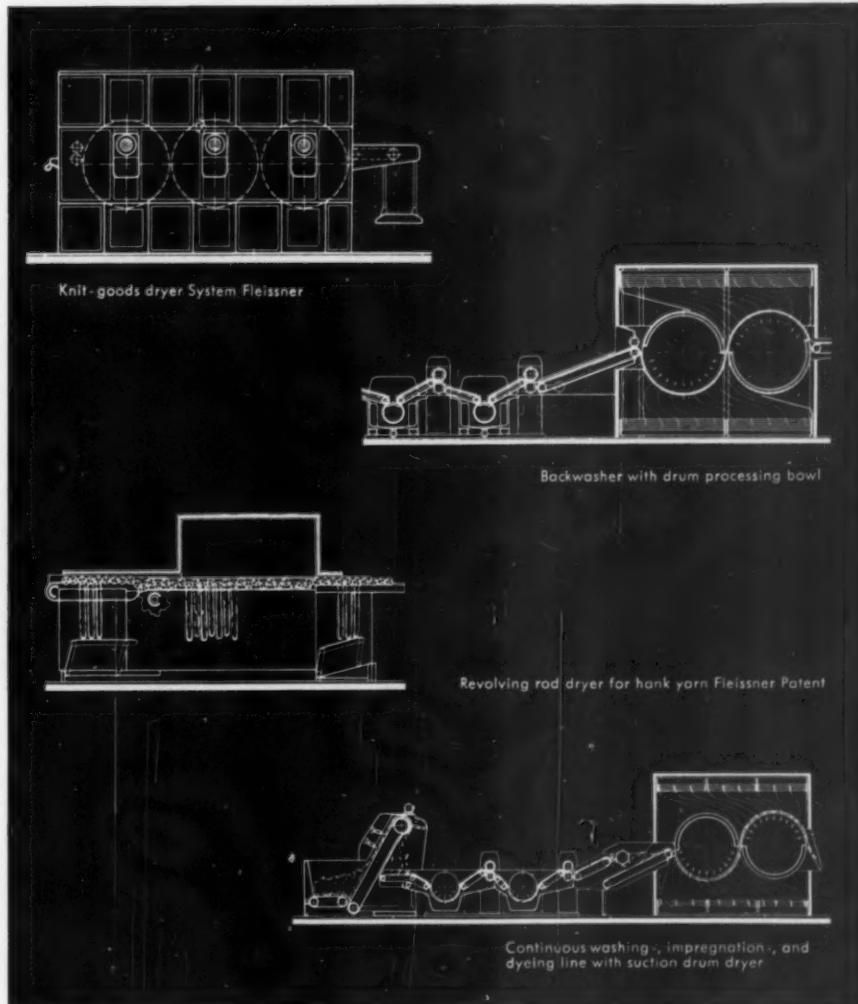
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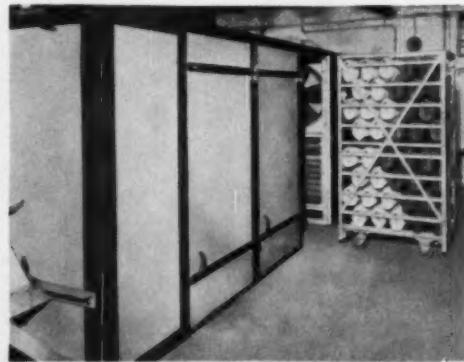


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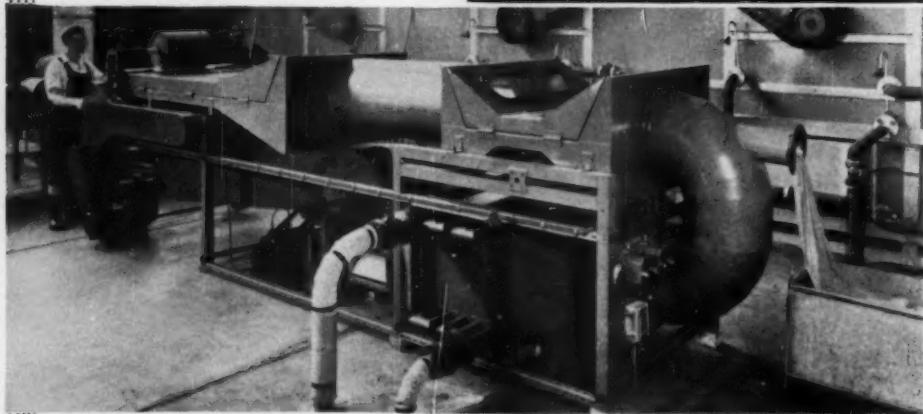


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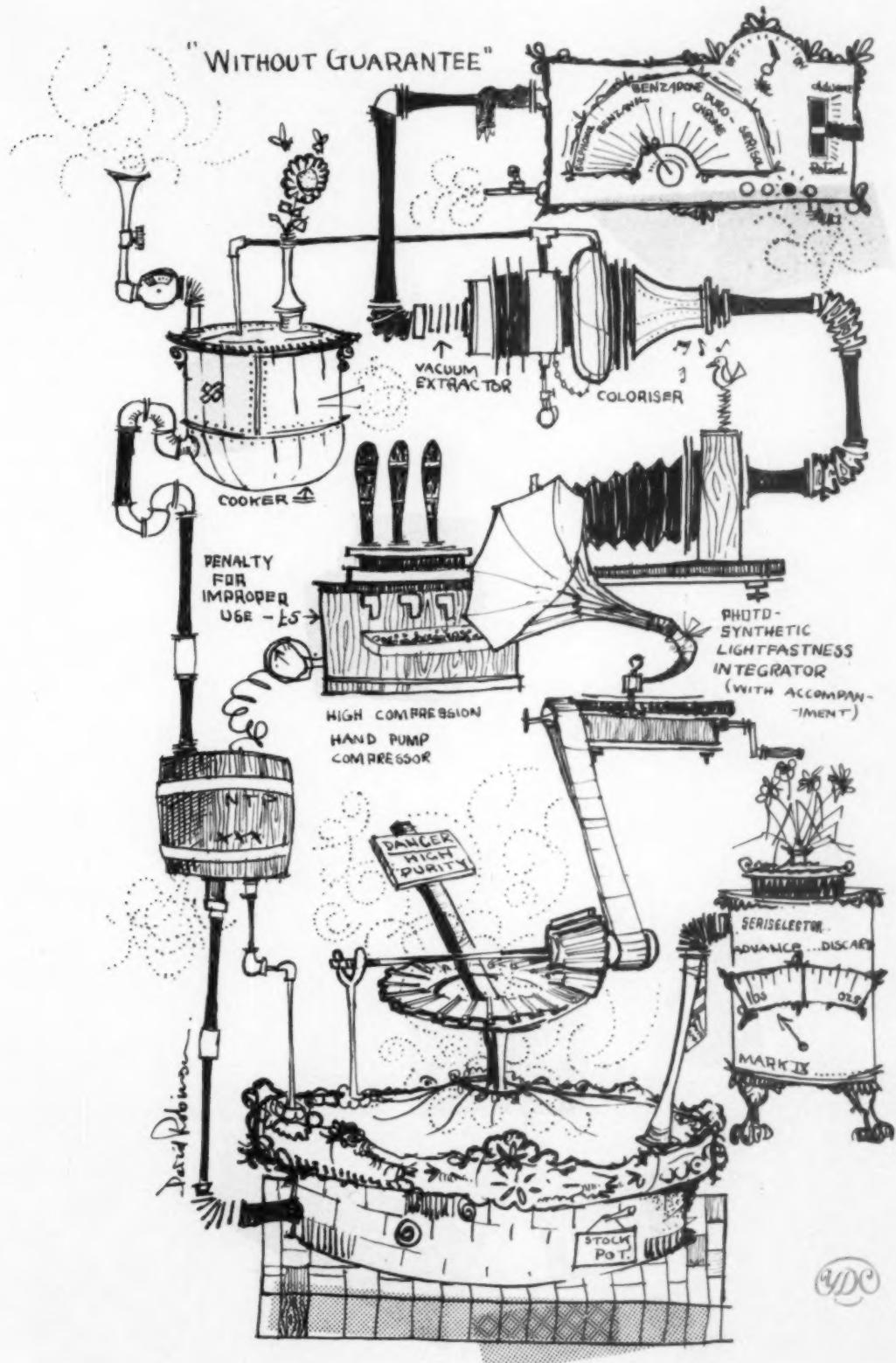
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G. S. J. WHITE

**European Free Trade Association—Origin Rules, Certification and Segregation of Stocks**

Pp. iii + 42. London: Association of British Chemical Manufacturers. 1960. Price, 3s. 6d. (2s. 6d. to members of A.B.C.M.).

During the past 2-2½ years, the Association of British Chemical Manufacturers has paid considerable attention to various problems which arose out of the concept of the European Free Trade Association and the Common Market policies, and during this time a high proportion of the time of several of its officers has been taken up in studies and discussions, both in this country and on the Continent, aimed at solving the problems relating to the interchange of goods between countries associated with one or other of the two groups.

One of the most difficult problems was that of safeguarding the trade of individual countries in E.F.T.A. from the effect of importation of low-price goods as a result of appreciable differences in tariff rates between countries both inside and outside the two groups. Means were therefore sought which would enable any particular chemical to be correctly and easily classified as to origin for the purposes of tariff procedure, and in the examination of this problem the A.B.C.M. played a very important part. During 1960, meetings of the Association were held at which the individuals and officers concerned explained to members the methods which were being put forward; at these meetings questions were invited and discussion

took place. The general consensus of opinion was that these discussions and meetings had been so useful that the publication of the reports and discussions might be of appreciable help to all the people in the industry who were concerned with both exporting and importing chemical products in the U.K.

As a result the Association has now published this 42-page booklet, which sets out, in the form of an introductory talk by Mr. W. A. M. Edwards, followed by six papers, the various aspects of the problems involved in operating the European Free Trade Association with respect to chemicals. Whilst the information contained in this book can by no means cover every aspect, it is lucidly set out and gives a remarkably clear explanation of some of the somewhat complex rules which have to be observed in dealing with the use and sale of chemical materials, and it will undoubtedly be of considerable help and use to anyone dealing in or concerned with the manufacture, supply, and sale of chemical products.

R. J. HANNAY

**Periodicals in the Chemical Society Library**

Pp. 48. London: The Chemical Society. 1960. Price, 5s. 0d.

This booklet shows the complete periodicals holdings of the Library of the Chemical Society, arranged alphabetically by the title as it appears on the title page of the periodical. Where necessary, cross references are made from all reasonable alternative forms of the title, and all publications of corporate bodies are shown both under the name of the body and under the title of the publication.

As members of the Society are entitled to borrow books from the Chemical Society's Library (see p. 3 of the January *Journal*) they should find this booklet of considerable value for periodicals which are either not taken or not retained by the Society.

M.T.

## Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.  
Any publication abstracted may be referred to by members of the Society on application to  
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**INDANTHREN DYESTUFFS "COLLOISOL"**—Fourteen Indanthren vat dyes previously marketed as highly dispersible powders under the designation "Stabilosol" have, from 1.1.61, been offered under the new name Indanthren Dyestuffs "Colloisol". There has been no alteration in either strength or particle size.

**FIRRAMOLL CW**—A softening agent of particular interest for application to yarns subsequently to be dried at high temperatures.

**SOLIDEGAL SR**—This is a levelling agent for vat dyeing. Its action is due to the formation of a leuco vat dye/Solidegal SR complex of great stability at low temperatures but which is very much less stable at normal dyeing temperatures. In consequence, with all but a few vat dyes, it is possible to effect uniform uptake of dye without loss of colour yield since the final exhaustion is hardly affected by the presence of the auxiliary product. In particularly difficult cases the levelling action of this product can be supplemented by adding a small amount of Solidegal GL. For maximum effect the dyebath should be set, at as low a temperature as possible, with the necessary amounts of caustic soda, hydrosulphite, and Solidegal SR, the finely dispersed unreduced vat dye pigment added and the liquor

circulated cold for 15-30 min. The temp. is then increased by 1 deg C/min to 40°C, then by 2 deg C/min to the maximum and dyeing continued for a further 15-30 min.

#### Ciba Ltd.

**CIBA AND CIBANONE DYES ON COTTON PIECE GOODS, VOLS. I AND II**—Volume II contains dyeings, in the majority of cases in three depths on mercerised cotton cloth, of 13 vat dyes belonging to the Ciba range of indigoid and related dyes and 80 Cibaneone anthraquinonoid dyes; these have been dyed by the batchwise exhaustion method. In addition, there are 12 dyeings of Ciba Blue 2RH and Ciba Blue GRH, alone and in mixtures, on unmercerised cotton cloth dyed by both pad-jig and pad-steam methods, and also dyeings on mercerised cotton cloth of 37 Cibaneone dyes in four depths dyed by the pad-steam method. Details of fastness characteristics, dyeing behaviour, marketed qualities, and recommended uses are appended alongside each set of dyeings. Volume I contains no less than 216 pp. of text and is, in fact, a complete manual covering every aspect of application of vat dyes to cotton cloth. Generous use is made of tables (21 in all) in the presentation of data, and there are 16 graphs covering the relation between caustic soda, hydrosulphite, and salt requirements and the quantity of dye being applied by each of the three main procedures. Important changes in the dyeing instructions for batchwise application include a reduction in the amount of hydrosulphite and caustic soda used in vatting and dyeing, and the recommendation that Cibaneone Blacks now be reduced in the dyebath, since the preparation of stock vat is not necessary. Further, the composition of the stock vat for dyes in groups CI, CII and CIII is now the same for each member. The stock vat method for applying the Ciba dyes is now known as CO; in this case the amounts of each ingredient recommended still vary from dye to dye.

#### Ciba Clayton Ltd.

**CIBACET ORANGE GF**—This disperse dye gives yellowish oranges on polyester and polyamide fibres and on secondary cellulose acetate and triacetate. It is particularly recommended where a high standard of fastness to hot pressing and pleating is demanded. On polyester fibres it may be applied at 125°C or at 100°C using o-phenylphenol as carrier. Fastness figures include: on polyester fibre—light 6-7 (½ standard depth), sublimation (60 s at 180°C) 4-5; on secondary cellulose acetate—light 7 (½ standard depth), washing (hand—40°C) 5, gas-fume fading 5, hot-pressing (120°C) 5.

#### Farbenfabriken Bayer A.G.

**ASTRAZON GOLDEN YELLOW GL**—This sheet is a supplement to pattern card No. Le782e, **ASTRAZON AND ASTRA DYESTUFFS FOR DIRECT PRINTING ON DRALON**.

**ACRAMIN MATT WHITE FKLR**—This is a white pigment based on rutile titanium dioxide for delustering synthetic fibre and regenerated cellulose woven materials using the Acramin FKLN process.

**ACRAMIN RED FRC**—This pigment gives bright bluish reds of very good fastness to light and to solvents when resin-bonded to the fibre. White discharges may be obtained on pad-dyed grounds using an alkaline discharge paste containing anthraquinone. Fastness figures for a direct print on cotton include—light (daylight) 7, hypochlorite bleach a 5, washing c (203°F) 5, perchloroethylene 4-5.

**BLANKOPHOR BA AND RA**—These fluorescent brightening agents are primarily intended for application to cellulosic fibres where treatment has to be carried out in hot liquors, e.g. in high-temperature bleaching of cotton with peroxide. They are highly substantive—indicated by the suffix "A"—and very good whites are obtained in absence of salt, even at the boil. They are also of interest on staple and continuous-filament viscose rayon, cotton-polyester fibre mixtures and silk. They are resistant to hydrosulphite but not to hypochlorite or chlorite bleach liquors, although their fastness to chlorine on the fibre is good. Fastness to light is of the same order as that of Blankophor BA.

**BLANKOPHOR REU**—This fluorescent brightening agent is a stilbene disulphonic acid derivative recommended for application to cellulosic fibres, polyamide fibres and their

mixtures. It is particularly suitable for single-bath application in finishing processes, even in presence of zinc salts.

**ISOLAN BROWN RLS: ISOLAN BROWN BL8: ISOLAN DARK BROWN RL**—These three new 2:1 dye:metal complex dyes give dyeings of excellent fastness on loose wool, slubbing, yarn, and cloth. They are also of interest on wool-cellulosic fibre mixtures, polyamide fibres and silk, and in vigoureux printing. Fastness figures on wool include—

	Light (daylight)	Milling (b)	Washing (c) (140°F)
Brown RLS	6-7	4-5	4-5
Brown BL8	7	4-5	4-5
Dark Brown RL	6-7	4-5	4

**RAPIDOGEN BRILLIANT BLUE N-IRA**—This stabilised azoic composition gives bright blues on cellulosic materials when printed and developed in either neutral or acid steam. Wet development is not recommended as insufficient yield of colour is obtained. Subject to approval, goods printed with this product and intended to withstand frequent laundering may be given the "Indanthren" label provided that requirements regarding the minimum depth of colour are satisfied. Fastness figures on cotton include—light (daylight) 6, washing c 4-5, peroxide bleaching 5.

**SIRIUS SUPRA LL DYESTUFFS**—This card contains dyeings in two depths on cotton cloth of 15 direct dyes, all characterised by the final suffix LL to denote outstanding fastness to light. Fourteen of these were previously included in the Sirius Supra range; one, Sirius Supra Grey B-LL, is a new product. This range is primarily intended for use on curtain and furnishing fabrics, and selections are offered for producing mixture dyeings of very good fastness on these materials.

**SUPRAMIN RED BBL**—This equalising acid wool dye gives bright bluish reds and is particularly recommended for use on ladies' dress materials and on hosiery and knitwear yarns. Particular features are the excellent fastness to light of the dyeings obtained, the very good solubility of this dye, and its ability to leave secondary cellulose acetate effects virtually unstained. Fastness figures on wool include—light (daylight) 6-7, perspiration 3, water (heavy duty) 4.

#### Farbwerke Hoechst A.G.

**REMAZOL TURQUOISE BLUE G**—This reactive dye gives very bright greenish blues when pad-dyed or printed on cellulosic materials. White discharges can be obtained on dyed grounds using White Discharge II. Fastness figures on viscose rayon crépe include—light 6, washing 95°C 3-4, chlorine 3-4.

#### Imperial Chemical Industries Ltd.

**DISPEROL FAST YELLOW T**—This homogeneous disperse dye is particularly recommended for dyeing polyester fibres, giving bright greenish yellows of very good fastness to light, even in pale depths. Greens of outstanding fastness to light may be obtained with it and Dispersol Blue TR. It is also of interest for dyeing polyester-cellulosic or polyester-wool mixtures, any cross-staining being readily removable by reduction-clearing. It is also useful for dyeing Acrlan, on which it builds up well. It may be direct-printed on polyester fibres by either steaming or dry-heat fixation, and is suitable for mélangeing. Dyed grounds are dischargeable to a good white. The appropriate supplement describing this dye has also been issued for insertion in the pattern card **DYESTUFFS FOR TERYLENE POLYESTER FIBRE (REF. 2996)**. Fastness figures on Terylene (dyed at 130°C) include—light (Bombay) 6-7, setting (15 s at 190°C) 4-5, washing (Test No. 3, once at 85°C) 5.

**PURE SCARLET CHROME FMS**—A molybdate lead chrome pigment of high opacity, soft texture and high stability to light, of interest in stoving finishes, paints, coating enamels, and for colouring plastics and mass-colouring linoleum.

**PURE SCARLET CHROME MS: SCARLET CHROME HMS**—A molybdate lead chrome pigment similar in properties and applications to Scarlet Chrome FM (above). The H-FM brand conforms to the 5% "soluble lead" requirement when tested according to B.S. 282:1953 Appendix K.

**PURE SCARLET CHROME LMS: SCARLET CHROME H-LMS**—A molybdate lead chrome pigment of particular value where high resistance to darkening and colour change on outdoor exposure is required. The H-LMS brand conforms to the 5% "soluble lead" requirements.

**PURE SCARLET CHROME LYS: SCARLET CHROME H-LYS**—A molybdate lead chrome pigment which, like Scarlet Chrome LM (above) is characterised by high resistance to darkening on outdoor exposure and is of particular value in industrial finishes where hiding power and weathering resistance are of major importance. The H-LY brand conforms to the 5% "soluble lead" requirement.

**PURE SCARLET CHROME YS: SCARLET CHROME H-YS**—A molybdate lead chrome pigment similar in properties and uses to Pure Scarlet Chrome LY (above).

**PROCINYL YELLOW G**—This leaflet is a supplement to **DYEING FOR TERYLENE POLYESTER FIBRE** (Ref. 2996). Procinyl Yellow G is an exception to the other members of the Procinyl range of reactive dyes for polyamide fibres in that it gives dyeings of outstanding fastness to heat treatments and washing and also, in full depths, to light on polyester fibres. It does not combine chemically with these fibres and for practical purposes may be treated as an ordinary disperse dye. Fastness figures on Terylene include—light (dyed at 130°C) 5–6, sublimation (Test A) 4–5, washing (Test No. 5, 5 × 30 min at 100°C) 5.

**DURAZOL BLUE RG**—This supplement to **DIRECT DYEING FOR STAPLE VISCOSITY RAYON** details the properties and uses of this direct dye. Fastness figures for a dyeing crease-resist finished with urea-formaldehyde include—light (Wilmslow) 6–7, washing (Test No. 3, once at 85°C) 3, perspiration 4–5 greener.

**BEDAFIL NL**—An anionic aqueous dispersion of a synthetic resin for filling light leather.

**CERRASOL AR**—An anionic product for lubricating fibres and yarns and for preventing soiling of viscose rayon tufted carpets.

**DISPERSEL CWL**—This is essentially a non-ionic product which, however, exhibits slight cationic properties and is intended as a levelling agent when applying milling acid dyes to wool yarn and cloth. It functions by reducing the rate of uptake of the dye by the fibre, but has only a slightly adverse effect on the final exhaustion of the dyebath. Addition of 1–5–3% of this product markedly improves the degree of levelness obtained, allows shorter dyeing times to be employed, and permits the use in mixtures of dyes previously considered to be incompatible. It does not cause precipitation of the dyes in the bath and does not affect the fastness properties of the dyeings obtained. It enables 1:1 dye:metal complex dyes to be applied in presence of 5% sulphuric acid instead of the customary 8% without sacrifice of level-dyeing behaviour. Used at a concentration of 3–6% (depending on the amount of acid present) it enables milling acid dyes to be applied from sulphuric acid dyebaths. Thus, when dyed from a dyebath containing 3% sulphuric acid, the level-dyeing assessment for Carbolan Brilliant Green 5G is 1 in absence and 5 in presence of 6% Dispersol CWL.

**TECHNICAL PUBLICATIONS: SUBJECT INDEX: SUPPLEMENT TO THE FIFTH EDITION—JANUARY 1959 TO JUNE 1960.**

**PROCION AND PROCION "H" DYEING FOR COTTON**—This card, together with **PROCION DYEING FOR VISCOSITY RAYON**, supersedes the earlier cards, **PROCION DYEING IN THE DYEING OF CELLULOSIC TEXTILES** and **PROCION "H" DYEING IN TEXTILE DYEING**. It contains jig dyeings in four depths on mercerised cotton cloth of 14 highly reactive cold-dyeing reactive dyes and 15 less reactive "H" brands. The 36 pages of text contain full details of recommended batchwise, pad-batch, and continuous dyeing methods, considerable use being made of diagrams in presenting the last-named. Time vs. exhaustion curves are given for each dye, in the case of the cold-dyeing dyes using both 5:1 and 30:1 liquor:material ratios. Repeated reference is also made to a manual, *Procion Dyeing in Textile Dyeing*, which will be issued shortly.

**PROCION DYEING IN TEXTILE PRINTING**—This card contains direct prints on mercerised cotton cloth of 13 Procion and 16 Procion "H" dyes produced using the Procion steaming process. The text includes a summary of

suitable printing processes, including the Procion-Resin resist process and other relevant information. For full details the user is referred to the manual, *Procion Dyeing in Textile Printing*, to be issued in the near future.

**PROCION DYEING FOR VISCOSITY RAYON**—This card contains dyeings in four depths on spun viscose rayon staple cloth of 14 "cold-dyeing" Procion reactive dyes and two "H" brands, Brilliant Blues H5G and H7G. This card, in conjunction with the manual, *Procion Dyeing in Textile Dyeing*, to be issued shortly, replaces the earlier cards and also T.I. (Dyehouse) No. 559.

**PROCION DYES ON SPUN VISCOSITY RAYON: WINCH DYEING**. The general presentation is the same as for the corresponding card for cotton referred to above. As in the latter, exhaustion curves are given for each dye applied under a variety of conditions, and the percentage fixation is also indicated.

**DYEING FOR MÉLANGE OR VIGOUREUX PRINTING WOOL AND TERYLENE**—This contains 18 examples of mélange printing on 70s combed white wool top and a further 13 on 4½-in. 3-den. Terylene staple top. The dyes used on the wool samples include acid, chrome, and reactive (Procion Black HG) dyes, disperse dyes being used on the Terylene. For wool, steaming for 2 h at atmospheric pressure with steam as moist as possible is recommended. For Terylene, steaming for 3 h at 5 lb/in<sup>2</sup> is satisfactory if Tunescal PH is added to the paste. Easier clearing of residual dye in the backwasher results from steaming for 45 min at 20–25 lb/in<sup>2</sup>; no carrier is required under these conditions.

**WOOL (PIECE): ACID DYEING**—This volume, the first of two, contains dyeings in four depths on wool cloth of 74 equalising acid dyes and 13 Ultralan 1:1 dye:metal complex dyes which, like the acid dyes, give level dyeings largely as a result of dye migration as dyeing is continued at the boil. The value of running the material in the blank dyebath to equalise the pH through the material is noted. A further recommendation based on recent experience with equalising acid dyes is that the temperature of the dyebath be raised to 50–60°C and held there until exhaustion is virtually complete; the temperature is then raised rapidly to b.p. and dyeing completed in the usual way. An even lower temperature is recommended where initial uptake of dye is particularly rapid, i.e. with carbonised material. This low-temperature exhaustion method results in the avoidance of gross initial unlevelness, with consequent reduction in the time required for levelling-out to take place during subsequent boiling.

#### TECHNICAL INFORMATION LEAFLETS

**Dyehouse No. 565 (Replaces No. 228). Affinity of the Water-soluble Dyes for China Clay**—Knowledge of the affinity of the dyes used for the clay is often of value in selecting dyes for colouring clay-filled papers. A method of determining affinity is described and results obtained with ranges of basic, acid, and direct dyes are tabulated.

**Dyehouse No. 581. Suitability of Direct Dyeing for the Dyeing of Viscose Rayon Cakes**—This supersedes the Technical Circular of the same title, issued in 1953. A test is described for assessing dyeing behaviour, and the range of direct dyes has been divided into four groups on the basis of the visual assessment of the degree of levelness obtained.

**Dyehouse No. 582. Procion Dyeing on Leather: Recent Additions to the Range**—Since the publication of T.I. (Dyehouse) No. 461, a further six highly reactive Procion dyes have been introduced; this leaflet describes their dyeing behaviour and fastness characteristics, together with those of the dyes previously presented.

**Dyehouse No. 584. 1961 Fashion Colours—Nylon Hose (The Midland Hosiery Dyers' and Finishers' Federation)**.

**Dyehouse No. 585. (Replaces No. 392). Dyeing of Terylene/Cellulosic Unions: Batch Dyeing Methods**—The main difference between this and the previous circular is that fuller information is given on the outlets for such mixture cloths when dyed with direct and disperse dyes; recently introduced dyes are included. Fastness characteristics are tabulated of disperse, vat, solubilised vat, and azoic dyes on Terylene-cotton, and also of disperse and direct dyes on Terylene-viscose rayon dyed by a one-bath process. Recipes for a range of typical colours on each blend are given, and the effect of resin finishing on the

hue and pleating fastness of disperse dyes on Terylene is appended.

*Dyehouse No. 586 (Replaces No. 409). Procion Dyestuffs: Application to Cotton and Linen by Batchwise Dyeing Techniques: Simplification of Dyeing Recipes*—The introduction of more cold-dyeing Procions and accumulation of experience of the range as a whole makes possible simplified recommendations as to dyeing temperature and alkali used. Sodium carbonate is now recommended as the most generally suitable alkali for batchwise dyeing of the cold-dyeing Procions between 20 and 40°C. The amount required is independent of dyeing temperature, but varies according to depth of colour and liquor: material ratio as set out in the accompanying table. Exceptions to these general rules are detailed.

*Dyehouse No. 588 (Replaces Nos. 403, 471 and 552). Procion Dyestuffs: Continuous Dyeing of Viscose Rayon Yarns and Loose Staple Fibre*—Recent experience in the continuous dyeing of viscose rayon yarn and staple is given, including continuous beam dyeing of yarn and simultaneous dyeing and sizing. On the basis of experiments to date, the continuous dyeing of cotton as loose stock and yarn is not recommended.

*Dyehouse No. 589. Dyeing of Terylene Polyester Fibre/ Wool Blended Materials: Supplement No. 3—Recipes for selected B.C.C. "Dictionary of Colour Standards" Shades*.

*Dyehouse No. 590. Pigments for Polypropylene Plastics*—The suitability of certain pigments for mass-colouring polypropylene has been examined. Those selected as being of interest are listed together with their fastness characteristics.

*Dyehouse No. 591. Terylene Polyester Fibre: Dyeing Recipes for Selected Shades from the B.C.C. "Dictionary*

of Colour Standards"—128 Recipes using disperse, diazotised, and developed disperse dyes, and azoic combinations are given.

*Dyehouse No. 595. Chrome Dyestuffs on Wool: Effect of Dispersol CWL on Level-dyeing Properties*—The level-dyeing behaviour of the range of chrome dyes applied by the afterchrome process has been determined at the acid-dyeing stage using the test described in T.I. (Dyehouse) No. 558. With the majority of chrome dyes, 1.5% of Dispersol CWL produces a significant improvement with no serious reduction in degree of exhaustion. Where the maximum amount (3%) is used, if the exhaustion is not good, chroming should be carried out in a fresh bath. With Solochrome Azurine B some dye-agent complex may be precipitated. In this case a small amount of Dispersol AC may be added to prevent this and the material give a rinse before chroming in a fresh bath.

### Sandoz Products Ltd.

**LANASYN BRILLIANT RED RL**—This neutral-dyeing acid dye gives bright reds of good fastness to light and wet treatments on wool and polyamide fibres. Like the other members of the Lanasyne Brilliant range, it is intended to supplement the Lanasyne range of 2:1 dye:metal complex dyes, with which it is compatible in dyeing behaviour and fastness properties. Fastness figures on wool include—light (daylight) 6, washing (60°C) 4-5, alkaline milling (strong) 5.

**LANASYN BROWN 2RBL**—This neutral-dyeing 2:1 (dye:metal) metal-complex dye gives reddish browns of good fastness on wool and polyamide fibres. Fastness figures on wool include—light (daylight) 6-7, washing (60°C) 4-5, alkaline milling (strong) 4-5.

## Abstracts from British and Foreign Journals and Patents

*The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

### I—PLANT; MACHINERY; BUILDINGS

#### Laboratory Dyeing Machine

I. Bucurehova and I. Bozaran

*Ind. Textild* (Bucharest), 11 (1960) 188-191  
Chem. Abs., 55 (23 Jan 1961) 2109

The machine consists of a water bath 20 x 7 x 8 in. with two rows of receptacles for beakers. Double walls lined with insulating material ensure minimum loss of heat. The patterns are stirred by up and down movement of the two rows of receptacles; speed of stirring can be adjusted between 30 and 60 strokes per min, to a distance of from 1-6 cm. Six 500-W heating elements can bring the bath to the boil in 30-40 min. The receptacles are exchangeable with frames for open-width dyeing when necessary.

C.O.C.

#### PATENTS

#### Wet Treatment of Warps

Garnett & Sons and G. H. Langley

*BP* 857,702 (13 June 1956)

When wet treating, e.g. sizing, an open-width warp it is passed over a roller that applies the liquid and then immediately into a drying chamber and thence on to a beam. Its path through the drying chamber is formed by vertical narrow spaces through which hot air is forced in separate streams over the warp to ensure quick drying.

C.O.C.

#### Machine for Continuous Wet Processing of Knitted Fabrics

A. W. P. Mackintosh

*BP* 857,225 (18 Mar 1958)

A series of vats has a pair of horizontal feed rollers above each vat. These rollers feed a continuous knitted fabric vertically downwards into the liquor in the vat. They are mounted in a carriage which moves to and fro over the vat so as to fold the web in a zig-zag fashion and free from tension on to the liquor. Means are provided above each vat to wet the fabric before it reaches the liquor. The width of the vats is adjustable to suit the width of the fabric being processed. The liquor is passed under pressure from one vat to the other in the same direction as the fabric. The bottom of the vat is curved so

that the pressure of the liquor ensures that the fabric is turned the right way up for being withdrawn vertically out of the bath.

C.O.C.

#### Regulating the Contact Pressure of Pressure Rolls in Impregnating Machines

Gebruder Sucker *RP* 858,668 (Germany 22 Mar 1956)

#### Multi-purpose Calender

McKiernan-Tervy Corp. *USP* 2,865,118 (29 Nov 1954)

The top and bottom companion rolls have a control for the pressure they apply and are connected by a gear train which can be readily changed to convert them from one type of treatment to another, e.g. from friction calendering to schreinering, embossing or the like.

*USP* 2,931,113 (12 May 1959)

A simplified machine of the above type is described which needs less overhead and floor space. There is more compact arrangement of component parts, better balance and smoothness of drive, and better accessibility of parts and conversion from one type of operation to another.

C.O.C.

#### Drying Machine for Tubular Knitted Fabric and the Like

Fitch Textile Co. *USP* 2,929,127 (1 June 1956)

Machine which continuously dries tubular fabric to the desired width, faster and with less consumption of power per unit length than previous machines.

C.O.C.

#### Multilayer Coating

Ilford *RP* 858,118 (6 July 1956)

Two or more liquid coating compositions are applied, e.g. to paper, simultaneously via a liquid-permeable block of small pore dimensions. This block smooths out any turbulence in the flow of the compositions, so that the liquids are deposited in superposed layers without any intermixing between adjacent layers.

C.O.C.

#### Colour Embossing Calenders

Dornbusch & Co. *BP* 858,923 (Germany 4 Mar 1958)

Printing ink is fed directly from an ink fountain to a screen roll having an elastically yielding jacket, excess ink being removed from the roll by a doctor blade. The



**Paint, Varnish, and Resin Stripper**

Diversey (U.K.) *BP 860,373* (U.S.A. 4 Nov 1958)  
Addition of 0.5-5.0% of monomethyl acid orthophosphate to methylene chloride greatly increases its stripping ability.

*BP 860,374* (U.S.A. 11 Dec 1958)

Benzene sulphonate acid and alkylbenzene sulphonate acids (preferably Alk of 1-12 C) can replace the orthophosphate.

C.O.C.

**Hydrazine and Hydrazine Hydrate as Reducing Agents for Sulphur Dyes**

Whiffen & Sons *BP 857,429* (1 July 1958)

Hydrazine and hydrazine hydrate are excellent reducing agents for sulphur dyes. Thus a stock solution is made up of Thionol Navy Blue VS (C.I. Sulphur Blue 1) (11 g), hydrazine hydrate (100% w/w) (3 ml), 62°Tw NaOH (1-3 ml), and boiling water (97 ml). This is diluted with water (45 water: 55 stock solution) to prepare a dyebath for cotton, dyeing being accomplished by slowly raising the temperature to 90°C while exhausting the bath by slowly adding anhydrous Na<sub>2</sub>SO<sub>4</sub>, washing off, and oxidising with perborate.

C.O.C.

**Reserving Agents for Acrylic Fibres**

Dow Chemical Co. *USP 2,931,694* (29 July 1958)

Fibres composed of a major proportion of acrylonitrile and a minor proportion of polymers or copolymers of *N*-vinyl lactams, *N*-vinyl-2-oxazolidinone and *N*-vinyl-3-morpholine are rendered resistant to dyes by application of 0.1-20.0% of their weight of polyphenolic polymers or condensates. Phenol-formaldehyde, bis-phenol-alkylidene polymers and phenol-divinylbenzene or phenol-di-isopropenylbenzene derivatives are particularly suitable.

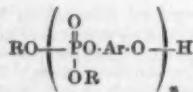
*USP 2,931,695* (29 July 1958)

**Compounds of formula—**

(each X = same or different Cl or Br; R = Cl, Br, F, OH, Alk of < 5 C, alkoxy of 1-4 C or halogenoalkyl of 1-4 C) are used. Examples are tetrabromo-*o*-cresol, tetrachloro-*o*-pyrocatechol and pentachlorophenol.

*USP 2,931,696* (29 July 1958)

Phosphorus-containing polyphenolic compounds of formula—



(Ar = a difunctional aromatic nucleus derived from hydroquinone, bisphenol-A, bisphenol sulphone or sulphoxide; R = same or different *pp'*-hydroxyphenyl, H or alkali-metal, and any combination or mixture of these may be present in the compound; n < 1), e.g. the product obtained from 5 mol. hydroquinone and 2 mol. phosphorus oxychloride, are used.

*USP 2,931,697* (29 July 1958)

**Polyphenolic alkanes of formula—**

(X, Y and Z = same or different H or *p*-hydroxyphenyl but there must be 3-6 *p*-hydroxyphenyl groups in the compound; n = 1-6), e.g. 1,1,2,2-tetrakis(4-hydroxyphenyl)ethane, are used.

*USP 2,931,698* (29 July 1958)

2,2'-Dihydroxy-5,5'-dichlorophenyl methane compounds or their homologues are used, e.g. Preventol GDC. C.O.C.

**Pentachlorophenol Emulsifiable Concentrates**

Armour & Co. *BP 858,687* (U.S.A. 20 June 1957)

A highly stable concentrate which disperses immediately when mixed with water to form emulsions of improved stability comprises pentachlorophenol (5-20% by wt.), a liquid aromatic hydrocarbon diluent (75-94) and, as

emulsifier, the disalicylate (2-3) of a compound of formula R-N(CH<sub>2</sub>CH<sub>2</sub>OH)CH<sub>2</sub>CHCH<sub>2</sub>N(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub> (R = aliphatic hydrocarbon of 8-22 C), e.g. *N*-tallow-*NN'*-tris(2-hydroxyethyl)trimethylenediamine disalicylate.

C.O.C.

**Aqueous Solutions of Formaldehyde-Urea Precondensates for Producing Resin Finishes Fast to Washing**

Toot Broadhurst Lee Co. *BP 857,283* (9 Mar 1956)

When diethylene triamine is used as the condensation catalyst, the stability of the undiluted solution increases as the proportion of the catalyst falls below 10%, so that when only 4% diethylene triamine is present the solution is stable for 2 days at room temperature. When an acid or potentially acid catalyst is added to the solution and it is used for finishing textiles, the resultant finish is faster to washing than the finish obtained with 40% diethylene diamine. As the amount of diethylene triamine present increases above 10%, so the proportion of the resin which is made fast to washing is rapidly reduced.

C.O.C.

**Resin Finish having Strong Resistance to Damage by Retained Chlorine**

Dan River Mills *USP 2,929,799* (14 May 1956)

The products obtained by reaction of 11-3 mol. of melamine with 1 mol. of ethylene urea readily produce stable methylol compounds when treated with HCHO at pH 7.0-8.5. These methylol compounds rapidly produce thermosetting resins when brought to pH < 7.0. They are used to produce crease-shedding textiles having strong resistance to damage by retained chlorine.

C.O.C.

**Resins for Treating Textiles, Paper, etc.**

Dow Chemical Co. *USP 2,930,716* (12 Oct 1956)

The adducts of HCHO with tetrahydro-5-hydroxy-2(1H)-pyrimidone (obtained by condensing 1,3-diamino-2-propanol with urea) are water-soluble but yield insoluble resins when heated, preferably at 325°F, for a few min. They impart excellent crease-recovery to cotton and the treated fabric has no tendency to absorb chlorine. Similar results are obtained on rayon, wool, cellulose acetate, nylon, and other synthetic-polymer fibres.

C.O.C.

**Water-repellent Aminoplastic Resinous Compositions for treating Textiles**

American Cyanamid Co.

*BP 858,860* (U.S.A. 12 Apr 1956)

An alkaline aqueous dispersion of (a) a compound of formula Y-NX-CR:Z (Y = H or Alk; Z = O or S; X and R = H, non-functional substituents or radicals containing Alk of > 6 C, either X or R must contain Alk of > 6 C), (b) 40-1000 parts (per 100 parts by weight of (a)) of an alkylated methylol melamine, an alkylated methylol urea, or a methylene alkylene urea, (c) 4-5-11.0 parts (per 100 parts of (a)) of an alkali-metal or organic amine soap, and (d) 10-35% (wt. of (a)) of a sorbitan or sorbitol fatty-acid ester, or alkylene oxide-alkylphenol condensate, fatty acid, the fatty acid radical being of > 6 C, is used to impart a durably water-repellent finish.

C.O.C.

**Fluorocarbon Acrylate and Methacrylate Esters and Polymers**

Minnesota Mining & Manufacturing Co.

*BP 857,689* (U.S.A. 23 Jan 1956)

The acrylate and methacrylate esters of *N*-alkyl, *N*-alkanol perfluoroalkanesulphonamides of formula R<sub>1</sub>SO<sub>2</sub>NR<sup>1</sup>R<sup>2</sup>CH<sub>2</sub>OH (R<sub>1</sub> = perfluoroalkyl of 4-12 C; R<sup>1</sup> = Alk of 1-6 C; R<sup>2</sup> = alkylene of 1-12 C) readily polymerise. When used as finishes on textiles, etc. they impart an oil- and water-repellent finish which is fast to repeated laundering and dry cleaning.

C.O.C.

**Fluorocarbon Acids and Derivatives—Oil- and Water-repellent Agents, Surfactants and Emulsifying Agents**

Minnesota Mining & Manufacturing Co.

*BP 857,335* (U.S.A. 29 Dec 1955)

Compounds of formula R<sub>1</sub>SO<sub>2</sub>NR<sup>1</sup>R<sup>2</sup>COOH (R<sub>1</sub> = perfluoroalkyl of 4-12 C; R<sup>1</sup> = H or Alk of 1-6 C; R<sup>2</sup> = alkylene bridge of 1-18 C) or their acid chlorides, metal and NH<sub>4</sub> salts, esters and amides, are useful for imparting oil- and water-repellency to textiles, paper, etc. In minute concentrations they can be used as wetting agents in aqueous and non-aqueous systems and as emulsifying and dispersing agents. Thus the Na salt of

$C_8F_{18}SO_2N(C_8H_5)CH_2COOH$  is an excellent wetting agent and its chrome complex is a good oil- and water-repellent agent (cf. *BP* 857,334 below).

*BP* 857,336 (U.S.A. 29 Dec 1955)

The vinyl and alkyl esters of the above acids can be used as surfactants. They and their polymers are excellent water- and oil-repellent agents of particular use on fibrous materials. Thus an aqueous dispersion of  $C_8H_{17}SO_2N(CH_3)CH_2COOCH_2CH_2$  yields an oil- and water-repellent finish on textiles which is fast to laundering and dry cleaning. C.O.C.

**Chromium Complexes of Fluorocarbon Acids for Rendering Paper, Textiles, Leather, etc. Oil- and Water-repellent**

Minnesota Mining & Manufacturing Co.

*BP* 857,334 (U.S.A. 29 Dec 1955)

The green water-soluble chlorine-containing chromium co-ordination complexes of perfluoroalkanesulphonamido-alkylenemocarboxylic acids, containing in the acid component a perfluorocarbon "tail" of 4–12 fully fluorinated atoms, e.g. *N*-ethyl-*N*-perfluoro-octanesulphonyl glycine,  $C_8F_{17}SO_2N(C_8H_5)CH_2COOH$ , when used as sizes for paper, impart both water- and oil-repellency. They can also be used on cellulosic films, textiles, leather, etc. C.O.C.

**Titanate-Silicone-Wax Compositions for Imparting Water Repellency**

General Electric Co.

*USP* 2,928,799 (13 May 1957)

A mixture of (i) 10–60% of a compound of formula  $Ti(OR^1)_4$  ( $R^1$  = aliphatic hydrocarbon of < 12 C or hydroxylated and aminated aliphatic hydrocarbon of < 12 and < 4 OH) or its aliphatic hydrocarbon-soluble partial hydrolysate, (ii) 25–75% of a methyl polysiloxane copolymer containing trimethylsiloxy and  $SiO_2$  units and 1–0.1–25  $CH_2$  per Si, (iii) 10–50% of a fluid organopolysiloxane of formula  $(R^2)_nH_2SiO(4-a-b)/2$  ( $R^2$  = unsubst. monovalent hydrocarbon;  $a = 1.0-2.25$ ;  $b = 0-1.25$ ;  $a + b = 1.96-2.25$ ), of viscosity 5 to 1,000,000 cS at 25°C, and (iv) a wax, renders porous materials water-repellent without requiring heat to develop the optimum water repellency. C.O.C.

**Magnesium Thioglycolate as Reducing Agent in Permanent Waving (X p. 233)**  
**Esters in Rosin Sizes (XI p. 234)**

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

**Studies on Colour and Constitution. I—Verification of Forster's and Knott's Rules**

B. Das, B. K. Patnaik, and M. K. Rout

*J. Indian Chem. Soc.*, 37 (Oct 1960) 603–610

To test the theoretical observations of Forster (*Z. Elektrochem.*, 45 (1939) 548) and Knott (*J.C.S.*, (1951) 1024), the following groups of spds. were prepared: (a) merocyanines, derived from pyrazolone, iso-oxazoline, and rhodanines as the acidic nuclei and 4-phenylthiazole as the basic nucleus; (b) *p*-dimethylamino-benzylidene deriv. of ketomethylene cpd.; (c) *p*-dialkylaminostyryl dyes; and (d) unsymmetrical cyanines; also, the *aza* analogues of (a), (b), (c), and (d). Forster's rule that "the absorption max. of the dye will increase with the decreasing tendency of the chain of chromophoric atoms lying between two auxochromes to take up the characteristic charge", and Knott's more empirical generalisation, which has its basis in the above, that "if the carbon atom at which replacement takes place is separated from the active auxochromic atoms by an odd number of conjugated atoms, a hypsochromic shift results, whilst separation by an even number of atoms results in a bathochromic shift", are found to be amply verified in the above cpd. H.H.H.

**Polarisation and Relaxation Effects in the Temperature Dependence of the Absorption and Fluorescence Spectra of Aromatic Compounds in Polar Solvents**

E. Lippert, W. Lüder, and F. Moll

*Spectrochim. Acta*, (1959) 858–869

*Chem. Abs.*, 54 (25 Mar 1960) 5243

With decreasing temperature the absorption bands are displaced to the red. The position of fluorescence maximum occurs at a temperature for which the relaxation

time of solvent dipole orientation agrees approximately with the lifetime of the excited state. C.O.C.

**Phytolacca decandra L. (Pokeberry). I—A Contribution to a Knowledge of the Betacyane**

H. Wyler and A. S. Dreiding

*Helv. chim. Acta*, 44 (Feb 1961) 249–257

The major red-violet pigment of the title substance, called phytolaccanin, has been isolated, crystallised, and its identity with betanin (the pigment of the red beet) established by analytical, spectrographic, electrophoretic, and chromatographic behaviour. Acid hydrolysis afforded a cryst. aglucone, identified as a mixture of betanidin and isobetanidin hydrochlorides. The pokeberry was also found to contain small amounts (< 5% in all) of three other pigments, viz., isobetanin, prebetanin, and iso-prebetanin. H.H.H.

**The Constitution of Suint. III—The Colouring Matter**

I. E. B. Fraser and E. V. Truter

*J. Textile Inst.*, 51 (Dec 1960) T 863–T 875

A partial formulation of the characteristic colouring matter of suint from normal sheep is given. The fundamental molecular unit is methyl 10-(2,5-dihydroxyphenyl)-decanoic acid, which is associated, usually non-stoichiometrically, with a nitrogenous  $\beta$ -diketone. The phenolic moiety undergoes oxidative polymerisation, and gives rise to brown or black pigments. E.V.T.

**Pyrazolone Dyes**

P. Ilinov and P. Kh. Vasilev

*Khim. i Ind. (Sofia)*, 32 (4) (1960) 115–117  
*Chem. Abs.*, 55 (23 Jan 1961) 2109

Fifteen azo dyes were produced by coupling 1-(4-sulphophenyl)-3-methyl-5-pyrazolone with— aniline, 2-aminophenol-4-sulphonic acid, 2-aminobenzoic acid, 5-amino-2-hydroxybenzoic acid, 3-nitro-4-aminotoluene, 4-chloro-2-aminophenol, 4-chloro-2-aminophenol-6-sulphonic acid, 2-amino-4-nitrophenol-6-sulphonic acid, 1-naphthylamine, 1-amino-2-naphthol-4-sulphonic acid, and 1-amino-8-naphthol-3,6-disulphonic acid. Coupling proceeded best at pH 7.0–7.5. C.O.C.

**Photoreduction of Janus Green B (C.I. 11050)**

R. Havemann, H. Pietsch, and H. Wielgoss

**I—The Proton Equilibria of Janus Green B and its Reduction Products**

*Z. wiss. Phot.*, 54 (1960) 91–99

Spectrophotometric analysis of the absorption of light by Janus Green B in aqueous solution as a function of pH indicates equilibrium between the unprotonated and protonated forms, in which, with decreasing pH, 1, 2, and 3 protons, respectively, are added. For the equilibrium between the unprotonated and singly protonated forms,  $pK$  is 11.74, and for that between the singly and doubly protonated forms, 1.76. Potentiometric titration with  $TiCl_3$  indicates reduction of the dye in two stages, firstly an irreversible uptake of 4 H atoms to yield *NN*-diethylsafranine and *p*-aminodimethylaniline, and then reversible formation of leucodiethylsafranine.

**II—Photoreduction of Janus Green B by Stannous Ion**

*Chem. Abs.*, 55 (23 Jan 1961) 1247  
*Ibid.*, 100–109

Photoreduction of the dye by  $Sn^{2+}$  induced by the light of a 150-W projection lamp, and followed spectrophotometrically at 595 m $\mu$  occurred in two steps. Firstly the light accelerates an irreversible hydrogemination fission, forming diethylsafranine and *p*-aminodimethylaniline, which also occurs in the dark. Kinetic analysis of the dark- and photo-reductions indicates that this step occurs through the intermediary of a 1:1  $Sn^{2+}$ : dye complex. Addition of a solvent of lower polarity (acetone) increases the rate of the dark- and photo-reductions, consistent with decreased dissociation of the complex and also with lowered energy of activation. The quantum yield for this stage is low, roughly about 0.01. In the second step, diethylsafranine is photoreduced to leucosafranine. A photo-stationary state results, when the rate of photoreduction equals the rate of thermal oxidation of the leuco compound to dye by  $Sn^{4+}$ . C.O.C.

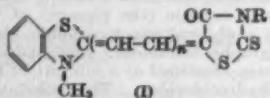
**Deviations of Merocyanines Derived from Benzo-thiazole and Substituted Rhodanines**

B. K. Sabata, B. K. Patnaik, and M. K. Rout

*J. Indian Chem. Soc.*, 37 (Oct 1960) 613–616

Mero-, merocarbo-, merodicarbo-, and merotricarbo-cyanines (I;  $n = 0, 1, 2, 3$ ) derived from substituted

rhodanines (as acid components) and benzothiazoles (as basic components) are described, and the deviations of absorption max. of individual members are calculated after taking into account the relevant data for the corresponding symmetrical cyanines and oxonols. The effect of increasing the chain length by addition of vinyl groups on the deviation in the above merocyanines is also determined—



H.H.H.

### Fungus Pigments. XII—Structure and Synthesis of Thelephoric Acid

J. Gripenberg

Tetrahedron, 10 (1960) 135–143

Chem. Abs., 55 (23 Jan 1961) 1565

Evidence showing that thelephoric acid is 2,3,8,9-tetramethoxybenzobis[1,2-b:4,5-b']benzofuran-6,12-quinone.

C.O.C.

### Biogenesis of Phenazine Pigments

R. E. Carter and J. H. Richards

J. Amer. Chem. Soc., 83 (20 Jan 1961) 495–496

It is suggested that biogenesis of pigments containing the phenazine nucleus, e.g. spirocyanine, chlororaphin, and iodinim, may occur through an oxidative dimerisation of anthranilic acid. Chlororaphin, extracted from cultures fed on anthranilic acid-1-<sup>14</sup>C(I) gave products which showed 21 ± 3% activity in the phenazine ring, demonstrating incorporation of (I) into chlororaphin. F.J.

### pH-Dependence of the Dark- and Photo-reduction of Thionine (C.I. 52025) by Sulphite Ion

H. Pietsch

Z. wiss. Phot., 54 (1960) 109–117

Chem. Abs., 55 (23 Jan 1961) 1247

Dark reduction of Thionine by excess aq. sulphite is first order in dye concentration and occurs only at pH > 5, the pH dependence of the reaction rate indicating that only  $\text{SO}_3^{2-}$  ions are effective. Light absorbed by the Thionine strongly accelerates the reduction, which now occurs rapidly at pH 5. As the rate of photo-reaction decreases with increasing pH, it is probable that the rate of photoreduction of Thionine by  $\text{HSO}_3^-$  ions exceeds that by  $\text{SO}_3^{2-}$  ions.

C.O.C.

### Synthesis of Murexide (C.I. 56085) and Composition of the Product of its Reaction with Ca

A. M. Lukin and I. D. Kalinina

Trudy Vsesoyuz. Nauch.-Issledovatel Inst. Khim. Reaktivov, (23) (1959) 63–66

Chem. Abs., 55 (23 Jan 1961) 1641

Murexide was synthesised from alloxanthin by a modified Piloty and Finch (Ann., 333 (1904) 22) method. Yield and purity of the product depended upon the concentration of  $\text{NH}_4^+$ . When at least three times as much  $\text{NH}_4^+$  was used as in the original method, the purity of the product was independent of the amount of the reactants. Murexide with Ca formed a complex  $\text{C}_8\text{H}_5\text{CaN}_3\text{O}_7$ .

C.O.C.

### Copper (O) Phthalocyanine

G. W. Watt and J. W. Dawes

J. Inorg. &amp; Nuclear Chem., 14 (1960) 32–34

Chem. Abs., 55 (23 Jan 1961) 1265

Reduction of Cu(II) phthalocyanine (C.I. 74160) with K in liquid  $\text{NH}_3$  gives dipotassium Cu(O) phthalocyanine anion complex with possible intermediate formation of the monopotassium Cu(I) phthalocyanine anion complex and its disproportionation.

C.O.C.

### PATENTS

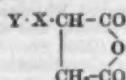
#### Colour Couplers

ICI BP 858,482 (14 Apr 1958)

Colour couplers of formula—

 $\text{A-NH-CO-CHR}^1\text{-CHR}^2\text{-COOH}$ 

(either  $\text{R}^1$  or  $\text{R}^2 = \text{H}$  and the other = XY (X = S or  $\text{SO}_2$ ; Y = Alk, cycloalkyl, aralkyl or Ar); A-NH = residue of a colour coupler containing an amino group) are obtained by treating the amino-containing colour coupler with an anhydride of formula—



They may consist of one of both of their two isomers. They are especially suitable for use in the silver halide emulsion layer of multilayer film or paper used for colour photography.

BP 858,564 (14 Apr 1958)

Modification of BP 830,797 (J.S.D.C., 76 (June 1960) 382), compounds of formula  $-\text{NH-CO-CHR}^1\text{-CH}_2\text{-CHR}^2\text{-COOY}$  (Y = H, Alk, cycloalkyl or aralkyl;  $\text{R}^1$  or  $\text{R}^2 = \text{H}$  and

the other  $\text{NR}^3\text{-acyl}$  or  $-\text{N}^{\text{CO}}\text{Z}$  ( $\text{R}^3 = \text{H}$ , Alk, cycloalkyl, aralkyl or Ar; Z = ethylene phenylene or tetrahydrophenylene; A-NH = residue of a colour coupler containing an amino group), being used as the colour couplers.

C.O.C.

### Metal(Cobalt)-complex Monoazo Dyes for Wool, etc.

BASF BP 860,356 (Germany 10 Aug 1957)

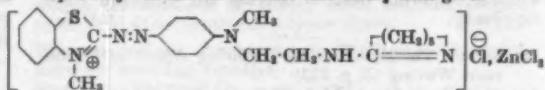
Diazotised *o*-aminophenols and *o*-alkoxyanilines free of  $\text{SO}_2\text{H}$  groups are coupled with *o*-coupling naphthols containing as sole substituents two groups  $-\text{SO}_2\text{NR}^1\text{R}^2$  ( $\text{R}^1 = \text{H}$ , Alk or phenyl;  $\text{R}^2 = \text{Alk or phenyl}$ ; or  $\text{R}^1$  and  $\text{R}^2$  with N may form a heterocyclic ring) and the monoazo compounds so formed are converted into Co-complexes which dye wool from neutral or weakly acid baths. Thus the Co-complex of the monoazo compound 4-chloro-2-aminopheno! → 2-naphthol-3,6-bis-sulphonmethylanilide is a reddish violet.

E.S.

### Monoazo Heterocyclic Basic Dyes

BASF BP 860,634 (Germany 4 Dec 1957)

Coupling components made by condensing lactim-O-alkyl ethers with amines are (1) coupled with diazotised 2-aminothiazoles or 2-aminobenzothiazoles and the products alkylated or aralkylated or (2) condensed with thiazolone-2-hydrazone or benzothiazolone-2-hydrazone alkylated or aralkylated in the 3-position as in BP 782,310 (J.S.D.C., 73 (1957) 526) to give red, violet, and blue basic dyes. Thus the condensate of caprolactim-O-methyl ether and *N*-methyl-*N*-m-tolylethylene diamine is condensed with 3-methylbenzothiazolone-2-hydrazone in presence of aq.  $\text{FeCl}_3$ . Addition of  $\text{NaCl}$  and  $\text{ZnCl}_2$  then gives—

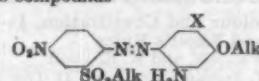


which dyes polyacrylonitrile reddish blue. E.S.

### Diazo Components for Azoic Greens and Browns

FH BP 860,995 (Germany 27 June 1956)

#### *o*-Aminoazo compounds—



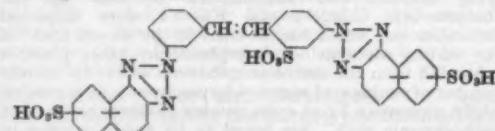
(X = Alk or OAlk), diazotised and coupled with arylides of 3-hydroxy-2-naphthoic acid, give greenish brown and green pigments and azoic dyes and prints on cellulose. Thus cotton impregnated with an alkaline solution of 3-hydroxy-2-naphtho-*o*-phenetidine is immersed in a solution of diazotised 2-amino-4,5-diethoxy-2'-methylsulphonyl-4'-nitroazobenzene to give a yellowish green.

E.S.

### Triazolyl Stilbene Fluorescent Brightening Agents

Gy BP 861,451 (Switzerland 24 Aug 1956)

#### Compounds of formula—

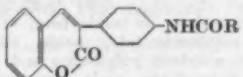


are fluorescent brightening agents for cellulose fibres. They have good affinity from aqueous solutions, particularly detergent solutions, and also in discharge and reserve printing pastes. They yield pure whites and better maximal brightening effect on cellulose than the comparable symmetrical bis(4,5-arylene-1,2,3-triazolyl-2)-stilbenes, and are faster to washing. An example is the tri sodium salt of 4,3'-bis-[5'-sulphonaphtho-1',2',4,5]-1,2,3-triazolyl-2-stilbene-2-sulphonic acid.

C.O.C.

**3-(*p*-Acylaminophenyl)coumarins—Fluorescent Brightening Agents for Polyacrylonitrile**  
FBY BP 862,286 (Germany 17 Dec 1958)

Compounds of formula—



(R = subst. or unsubst. Alk) are fluorescent brightening agents having affinity for polyacrylonitrile from solution or dispersions or by being added to spinning solutions or moulding powders of polyacrylonitrile. They are exceptionally resistant to chlorine, and acrylic fibres treated with them can be further brightened by chlorite bleaching. Thus the product in which R = CH<sub>3</sub> is added to a polyacrylonitrile solution from which filaments are then made. These are then chlorite bleached. The resulting filaments are outstandingly brightened and the brightening effect is very fast to light. C.O.C.

**Acedianthrones—Vat Dyes Yielding Brown Prints on Cellulosic Fibres**

Ciba BP 859,256 (Switzerland 4 Oct 1956)

Acedianthrone, 2,2'-dichloroacedianthrone, 2,2'-dimethylacedianthrone, or a bromine substitution product thereof, and 2,2'-diphenylacedianthrone are brown vat dyes which, while yielding only very weak dyeings, do yield strong prints on cellulosic fibres. C.O.C.

**Dye from Crude Perylene-3,4,9,10-Tetracarboxylic Acid Di-imide**

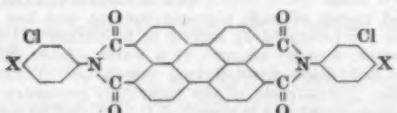
BP 861,218 (Germany 29 Sept 1956)

A paste of crude perylene-3,4,9,10-tetracarboxylic acid di-imide (I) is refluxed (optionally at reduced pressure) in an organic diluent of b.p. > 120°C with removal of components of b.p. > 100°C until the b.p. of the diluent is reached. The product has high tintorial strength, brilliance, and purity, and is used as a bluish red or maroon pigment for printing inks, lacquers, plastics, rubber, and spinning solutions. It may be obtained by melting naphthalene-1,8-dicarboxylic acid and acenaphthenequinone mono- or di-oxime with KOH or by condensing the tetracarboxylic acid or anhydride with NH<sub>3</sub>, and finally preparing a finely divided paste by conventional methods.

A.T.P.

**Pigments from Perylene Tetracarboxylic Acid**  
FH BP 860,889 (Germany 4 May 1956)

Red pigments for printing inks, lacquers, plastics, and spinning solutions are obtained by condensing perylene-3,4,9,10-tetracarboxylic acid or anhydride with 3-chloro- or 3,4-dichloroaniline, *viz.*—



(X = H or Cl).

A.T.P.

**Conditioning of Crude Polychloro Copper Phthalocyanines**

General Aniline USP 2,930,706 (11 Feb 1957)

Modification of USP 2,716,649 (J.S.D.C., 72 (1956) 394). The crude product is brought into finer physical form and to a more yellowish green by adding a water-insoluble benzeneid neutral liquid into the mass being milled. The addition is made at any time during milling before the mass is diluted.

C.O.C.

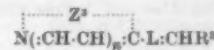
**Dicarbocyanine Dyes**

Kodak BP 858,062 (U.S.A. 14 May 1956)

Dyes of formula—



(R<sup>1</sup> and R<sup>2</sup> = subst. or unsubst. Alk; n = 0 or 1; X = acid radical; Z<sup>1</sup> = atoms to complete a 5- or 6-membered nucleus; Z<sup>2</sup> = atoms to complete a benzoxazole, naphthoxazole, benzoselenazole, naphthoselenazole, benzothiazole, or naphthothiazole nucleus) have their photosensitising power much increased in presence of a base of formula—



or

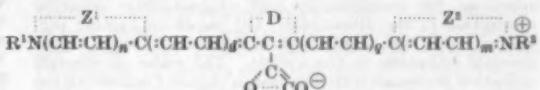


(R<sup>2</sup> = subst. or unsubst. phenyl; L = N or CR<sup>4</sup> (R<sup>4</sup> = H, cyano or alkoxy); Z<sup>2</sup> = atoms to couple a nucleus containing 1 or 2 N atoms. C.O.C.

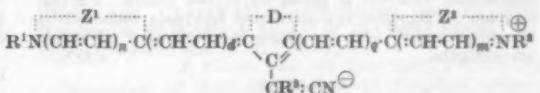
**Cyanine Dyes**

Kodak BP 862,445 (U.S.A. 13 Sept 1956)

Dyes of formula—



or

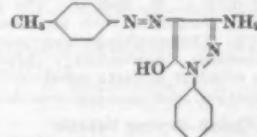


(R<sup>1</sup> and R<sup>2</sup> = Alk, subst. Alk, aralkyl or Ar; R<sup>2</sup> = cyano or alkoxy carbonyl; d, m, n, and q = 0 or 1; D = atoms to complete a cycloalkene ring of 5-6 C; Q, L = atoms to complete a 5- or 6-membered heterocyclic ring), e.g. 1,3-diethyl-5-[2,5-di(1-ethyl-2-naphtho[1,2]thiazolylidene)-cyclopentylidene]barbituric acid, have photosensitising properties. C.O.C.

**Dyes having Photographic Developing Properties**  
International Polaroid Corp.

BP 857,879 (U.S.A. 5 July 1957)

Dyes of formula [Y-S-R<sup>1</sup>-CO-NR<sup>2</sup>]<sub>n</sub>X (Y = 2,5-dihydroxyphenol, which may be substituted by Hal and Alk; R<sup>1</sup> = alkylene or arylene; R<sup>2</sup> = H, Alk, or Ar; n = 1 or 2; X = residue of an azo or anthraquinone dye and containing at least one reactive amino group, e.g.—



are "dye developers", i.e. they have the properties of both dyes and developing agents, having a wide range of uses. C.O.C.

**Water-dispersible Carbon Black (C.I. Pigment Black 6 and 7)**

Société d'Etude des Industries du Pétrole au Port de Strasbourg BP 862,018 (France 16 Jan 1957)

The pigment is treated with an aqueous oxidising solution of HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, KMnO<sub>4</sub>, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, NaClO<sub>4</sub>, or persulphuric acid at 20°C to b.p. The solution is of such strength as to provide the final product with a volatile content of < 10%. The volatile content is the oxygen and hydrogen chemically combined with the pigment which may be driven off by heating at 1750°F. This renders the pigment hydrophilic. C.O.C.

**Rendering Pigments Readily Dispersible in Organic Vehicles**

Armour Chemical Industries BP 860,868 (9 Oct 1956)

Treating pigments with a compound of formulas R<sup>1</sup>-NR<sup>2</sup>-R<sup>3</sup>-COOY (R<sup>1</sup> = Alk of 8-20 C or Alk subst. by amine or polyamine groups; R<sup>2</sup> = H or Alk; R<sup>3</sup> = chain of 1-6 C; Y = H, metal, alcohol or acyl) is used. Thus Prussian Blue (C.I. Pigment Blue 27) is treated as an aqueous slurry with N-alkyl-β-aminobutyric acid derived from coconut oil for 1 h. After filtering, drying, and grinding, a pigment is obtained which disperses readily in an organic vehicle. C.O.C.

New American Products (III p. 219)

Spectral Reflection of White Pigments (V p. 224)



top coating to wood treated with a polyvinyl acetate emulsion paint. After 9 months' outdoor exposure the films were entirely satisfactory and had not lost in gloss.

C.O.C.

### Coating Metal with Thermoplastic Synthetic High Polymers

Stahl- und Walzwerke Rasselstein/Andernach

BP 860,313 (Germany 13 Dec 1957)

The polymer of particle size  $> 100\mu$  is dispersed in an organic solvent which is immiscible or only partly miscible in water and whose b.p. is above the softening point of the polymer. The dispersion is then applied to metal surfaces and heated for a long time at a temperature about the softening point of the polymer and then dried by heating for a short time at a higher temperature. This enables thin ( $< 20\text{ g/m}^2$ ) non-porous coatings to be obtained.

C.O.C.

Cellulose-*p*-xylyl-bis-ether—a Yellow Compound having Green Fluorescence (VI p. 226)

Design of Unsaturated Polyester Resins for Surface Coatings (XIII p. 234)

## VI—FIBRES; YARNS; FABRICS

### Textile Properties of Corval and Moynel Fibres

A. F. Cheatham

Candian Textile J., 77 (23 Dec 1960) 55-65

Corval and Topel are crosslinked viscose staple fibres, Corval II and Topel II being newer forms of higher stability. Moynel is a staple fibre of high wet modulus. The properties, processing (including wet processing, dyeing, and finishing) and end uses of these fibres are discussed, with numerous graphical illustrations. Processing is similar to that of viscose rayon and/or cotton, the inherent fibre rigidity (and low elongation of Moynel) being allowed for. The fibres may be mercerised and given crease-resist finishes; the crosslinked fibres require little resin.

W.P.M.

### Bacterial Discoloration of Wool

I. E. B. Fraser and E. V. Truter

J. Textile Inst., 51 (Dec 1960) T 857-T 862

The factors that might explain why only some wools are stained green by the bacterial metabolite pyocyanin have been investigated. Bacteriological experiments show that there are no differences in the abilities of normal fleece constituents of immune and susceptible sheep to support pigment production. It is concluded that discoloration will occur only if the fleece contains adventitious bacterial nutrients which arise through damage to the hypersensitive skins of susceptible sheep.

E.V.T.

### Spectrophotometric Study of Yellowing in Wool Fabric

F. G. Lennox

J. Textile Inst., 51 (Dec 1960) T 1193-T 1209

Yellowing of wool fabric was studied by the differential spectrophotometric method. Yellowing occurred more slowly on heating in air than in aqueous solution. On heating in buffer solutions, yellowing increased with pH, but was also greatly influenced by the nature of the buffer ions. At pH 9, glycine caused least yellowing and pyrophosphate most. Hydrothermal yellowing was partly prevented by pretreatment with 0.1-M KCN. When exposed to ultraviolet radiation, wool yellowed rather more rapidly in dry than in moist air. A greenish colour developed on irradiation under anhydrous conditions, but disappeared on exposure to moist air, the colour reverting to the usual yellow. The optical density at 3200 Å due to irradiation increased as rapidly in wool that had been dyed or treated with a fluorescent brightening agent as in the untreated material. The yellow colour produced either by heating in pH 8 buffer, or by irradiation was partly discharged by treatment in dil. acid. Evidence is presented which suggests that cysteine is the amino acid chiefly responsible for the discoloration.

W.K.R.

### Mechanical Properties of Set Wool Fibres and the Structure of Keratin

M. Feughelman

J. Textile Inst., 51 (Dec 1960) T 589-T 602

Load-extension curves in water at 20°C of wool fibres

which had been strained to varying extents in boiling water and released in water at various temperatures for 1 h, show considerable modifications of the initial portion of the curve for untreated fibres. The degree of modification depends on the setting strain, the time for which the fibre was held at this strain, and the temperature at which it was released after setting. The modifications are explained in terms of a series zone model for keratin.

W.R.M.

### Setting Properties of Keratin Fibres in Relation to Structure

J. L. Stoves J. Textile Inst., 51 (Dec 1960) T 603-T 612

When set at zero extension in borax solution fibres from the skunk, Russian hare, kolinsky, and mink do not contract in boiling 5% metabisulphite solution, but those from reindeer, whitecoat, and Chungking pig contract. Hydrogen-bond formation does not play a significant part in such setting of fibres from mink, skunk, or whitecoat, but fibres from Russian hare, kolinsky, reindeer, and Chungking pig contain more hydrogen bonds than normal fibres. Linkages other than hydrogen bonds are responsible for the resistance of skunk and mink fibres to metabisulphite setting.

W.R.M.

### Effects of Chemical Modifications on Physical Properties of Wool: A Model of the Wool Fibre

W. G. Crewther and L. M. Dowling

J. Textile Inst., 51 (Dec 1960) T 775-T 791

Conversion of disulphide bonds to *S*-methyl groups accelerates supercontraction in LiBr solutions. Evidence is presented that the rate of supercontraction is partly determined by interchange reactions between thiol and disulphide groups and that inhibition of supercontraction in the presence of free bromine is due to oxidation of thiol groups. It is concluded that contractile structures are not crosslinked by disulphide bonds but are embedded in material containing inter- and intra-molecular disulphide bonds (the matrix), the rate of plastic deformation of which is affected by thiol interchange with disulphide bonds. Nitration, iodination, mild oxidative treatments, and acids and alkalis accelerate the first stage of supercontraction in 6-M LiBr at pH 6, but retard the second stage. This retardation does not occur in 4-M LiBr/1-N HCl, suggesting that ionic interactions are responsible for retardation. The results are explained in terms of a model assigning low-sulphur proteins to microfibrils and high-sulphur protein to the intermicrofibrillar matrix.

W.R.M.

### Nuclear Magnetic Resonance of Water Sorbed in Fibrous Materials

T. M. Shaw, R. H. Elskens, and R. E. Lundin

J. Textile Inst., 51 (Dec 1960) T 562-T 572

The sorption of water in wool, silk, and nylon fibres, and in wool cortical cells, has been studied by NMR, and proton resonance line shapes and relaxation times have been determined as functions of moisture content. The width of resonance depends on both sorbent and moisture content, and decreases as moisture content increases. It is suggested that water is bound in clusters to the sorbent and that surface migration is the dominant mode of magnetic relaxation.

W.R.M.

### A Generalised Theory for Combination of Wool Keratin with Acid or Alkali

L. Peters J. Textile Inst., 51 (Dec 1960) T 1290-T 1301

A generalised Donnan theory, assuming ions to be adsorbed at sites, is developed to describe the titration curve of an insoluble protein which possesses unequal numbers of carboxyl and amino groups. Equations derived apply to combination with acid or alkali and include a treatment of the isoelectric point. From the constancy of the isoelectric pH of keratin it is concluded that  $K^+$  and  $Cl^-$  do not combine with keratin.

S.B.D.

### Absorption of Acids by Wool from Solutions Containing Organic Solvents. I—Absorption of Hydrochloric Acid from Aqueous Solutions of Methanol and Dioxan

P. Larose and R. Donovan

J. Textile Inst., 51 (Dec 1960) T 1302-T 1310

Absorption of HCl by wool from aqueous soln. containing 24, 40, and 80% by weight of methanol, and soln. with 45 and 82% of dioxan was measured at 22°C. Titration curves were similar to those obtained with aqueous HCl, but displaced a distance which depended on the change of dissociation constant of the carboxyl group.

caused by change of medium. With certain limitations, the shape of the titration curves can be explained either by the Gilbert-Rideal theory or by the Domian equilibrium theory. S.B.D.

**Diffusion of Some Sulphonic Acids in Keratin in Presence of Water and Alcohols**

J. A. Medley and D. K. Ramsden

*J. Textile Inst.*, 51 (Dec 1960) T 1311-T 1322

Transfer rates of a series of alkylbenzenesulphonate ions between aqueous soln. and horn keratin have been measured by radio-tracer techniques. Rates are increased by addition of n-propanol and n-butanol to the external soln., effects being greater for larger alkyl groups and for higher concn. of ion in the keratin. Evidence is presented that transfer rates, whether alcohol is present or not, are governed by Fickian diffusion within keratin. Butanol is preferentially absorbed, but it appears that the resulting acceleration of organic ions is not due to swelling action. An explanation is proposed in terms of hydrophobic interactions, on which the affinity of an ion depends. When the solvent is water alone, these interactions immobilise the ion on the keratin structure, and the fraction of ions remaining free is inversely related to affinity. With mixed solvents, interactions are possible between free ions and alcohol, which increases the fraction of ions free. S.B.D.

**Action of Heat on Wool**

J. W. Bell, D. Clegg, and C. S. Whewell

*J. Textile Inst.*, 51 (Dec 1960) T 1173-T 1182

When wool is heated in air for different times at 200°C it becomes first yellow and eventually brown. Water- and alkali-solubilities increase, and cystine and tyrosine contents fall. Resistance to extension is greatly reduced. There appears to be little attack on the amino groups and little change in the salt linkages, but the base-combining power is increased. The most noticeable chemical change is the gradual fall in cystine content to almost nil. Wool modified by sulphonation, particularly by immersion in conc.  $H_2SO_4$ , for 15 s followed by thorough rinsing, or treated with aluminium sulphate, with a mixture of borax and boric acid, or with a mixture of THPC and urea, is less susceptible than untreated wool to discolouration when heated at 200°C. W.K.R.

**Effect of Heating Wool Containing Alkali**

G. P. Norton and C. H. Nicholls

*J. Textile Inst.*, 51 (Dec 1960) T 1183-T 1192

Yellowing caused by drying wool containing alkali at high temp. is shown to be dependent on the time and temp. of drying and the presence of strong alkali. The presence of mild alkali ( $Na_2CO_3$ ) in quantities likely to occur in industrial processing did not produce yellowing additional to that shown by drying alkali-free wool under the same conditions. The discolouration was partly discharged by acid, it faded rapidly in air, and the wool was readily bleached by a mild hydrogen peroxide treatment. Yellowing was not prevented by the addition of oxidising or reducing agents to the alkali, or by chemical modification of the wool. It is suggested that, under mild conditions, discolouration is due to cystine breakdown, but when there is more intense discolouration, other amino acids, such as tyrosine, may be involved. W.K.R.

**Effect of Ionising Radiation on Strength and Extensibility of Wool**

G. Satlow *Melland Textilber.*, 41 (Dec 1960) 1525-1527

Wool fibres irradiated with varying (0.2-5.0  $\times 10^6$  rads/cm<sup>2</sup>) doses of thermal neutrons have reduced wet tensile strength and extension at break. The stress-strain curves are shorter and run roughly parallel to but lower than the curves of the untreated fibre. The effect increases with the intensity of irradiation and is similar to the effect of prolonged exposure to weather. W.M.

**Structure of Frizzle Mutant Feather Keratin**

S. Krimm *J. Mol. Biol.*, 2 (1960) 247-249

*Chem. Abstr.*, 55 (9 Jan 1961) 635

The X-ray diffraction patterns of normal and homozygous feather keratins are distinctly different, while that of heterozygous keratin is roughly intermediate between the other two. The mutant keratin shows a much higher proportion of disordered material and the crystalline component is less well oriented than in normal keratin. The mutation is probably correlated with a change in the

amino-acid composition of the polypeptide chains. By a solubilisation method a disordered  $\beta$ -keratin (according to its X-ray diffraction pattern) can be removed from feathers and comprises 35% of normal, 50% of heterozygous, and 75% of homozygous feather. Polarised infrared spectra of feather show significant reduction in the dichroism of NH and CO stretching bands, at 3240 and 1650 cm<sup>-1</sup> respectively, in the mutant as compared with that in normal feather. C.O.C.

**Studies on Rabbit Fibres. XXXVII—Relations Between Feltability and the Carrotting Factors**

S. Okajima, S. Ikeda, and T. Dote

*J. Soc. Textile Cellulose Ind. Japan*

16 (Nov 1960) 975-980

Relations between the density of the felt prepared from carrotted Angora rabbit furs and the carrotting factors, i.e. concentration of carrotting reagents ( $Hg(NO_3)_2$ ,  $HNO_3$ , and  $NaNO_3$ ) were studied by a statistical technique. The concentration of  $NaNO_3$  is significant, but other factors are not significant at a 5% level. W.P.M.

**Influence of Spinning Conditions on Textile Properties of Man-made Fibres**

A. B. Pakshver

*Faserforsch. und Textiltech.*, 12 (Jan 1961) 9-18

The influence of different arrangements of the polymer chains on fastness and sorption of different chemicals is discussed, and the importance of interchain attraction is stressed. Different properties of man-made fibres are primarily a consequence of differing time factors in extrusion. Fibre characteristics, especially dye sorption, can be varied by altering extrusion and spinning conditions. Some suggestions are made regarding the production of fibres with particular properties. W.R.M.

**Polyamide Fibre Damage by Peroxate Solutions**

A. Engeler and W. Schefer

*Textil-Rund.*, 16 (Jan 1961) 4-10

A determination of the damage to nylons 6, 6.6, and 6.10, caused by boiling sodium perborate solutions, showing the effect of filament size, delustring agents, and chromium or manganese salts on the rate and extent of oxidation. Results are expressed in terms of loss of tensile strength, breaking extensibility, and viscometric molecular weight, and an increase in amino and carboxylic end-groups. Determination of the rate of decomposition of perborate and the influence on this of sodium phosphate, soap, soda, etc., are described. P.B.S.

PATENTS

**Cellulose-p-xylyl-bis-ether—a Yellow Compound having Green Fluorescence**

Rayonier

BP 861,430 (U.S.A. 1 Apr 1959)

Cellulose activated with liquid ammonia, treated with metallic sodium, and then etherified with  $\alpha$ -dibromo-p-xylene yields a product which is brilliant yellow and has strong greenish fluorescence in ultraviolet radiation. It may be xanthated, formed into viscose, and spun into acid to yield filaments possessing the original brilliant fluorescence. These filaments may be blended with other fibres to give an appearance of greater whiteness to the blends. Other uses include the application of this cellulose ether to textiles to yield fluorescent products, and to the manufacture of luminous paints, plastics, pigments, and inks. C.O.C.

**Alkaline Treatment for Facilitating the Processing of Polyester Fibres and Yarns**

N.V. Onderzoeksinstiitut Research

BP 859,220 (Holland 14 Dec 1956)

Treatment, preferably at  $< 60^\circ C$ , with an aqueous solution of an alkali-metal hydroxide and an anionic surfactant, facilitates the drawing, spinning, winding, etc., of polyester fibres and yarns. When the fibres are heat stabilised, this alkaline treatment follows heat stabilisation. C.O.C.

**Acid Treatment for Imparting Increased Affinity for Anionic Compounds to Acrylic Fibres**

Chemstrand Corp.

USP 2,932,550 (14 Oct 1953)

Acrylic fibres containing  $< 80\%$  acrylonitrile and 2-10% of an N-heterocyclic monomer when treated for 1-60 s with 1-10% aqueous acid at 100-212°F and then rinsed free of excess acid dye readily with acid or direct dyes in a neutral bath. C.O.C.

**Ion-exchange Fibres, Films, and the Like**

Rohm & Haas Co. *BP 858,864* (U.S.A. 29 May 1956)  
 Fibres, films, etc., having at least one dimension of 0.1–20.0 mils and made from a crosslinked linear addition polymer containing on the number of recurring units < 7% of ion-exchange groups and < 0.5% of cross-linkages have an ion-exchange capacity of 0.3–8.0 milliequivalents per g. Several methods of producing such polymers are described.

C.O.C.

**Alkoxyalkylvinyl and  $\alpha$ -Methylvinyl Sulphide Polymer Fibres, Films, etc.**

Rohm & Haas Co. *BP 858,865* (U.S.A. 29 May 1956)  
 Thermoplastic addition polymers derived from ethylenically unsaturated molecules comprising < 0.2% by weight of a compound of formula  $\text{CH}_2\text{CR}^1\text{S}-\text{R}^2\text{OR}^3$  ( $\text{R}^1 = \text{H}$  or  $\text{CH}_3$ ;  $\text{R}^2 = \text{CH}_2$ ,  $\text{CH}(\text{CH}_3)$  or  $\text{C}(\text{CH}_3)_2$ ;  $\text{R}^3 = \text{Alk}$ ) are suitable for forming into fibres, films, etc.

C.O.C.

**The Constitution of Sint. III—The Colouring Matter**

(IV p. 221)

**Permanent Setting of Wool** (X p. 231)**Adhesion of some Elastomers to Fibres and Foil of Viscose Rayon Modified with Methyl and Isopropyl Isocyanates** (XIII p. 235)**VII—DESIZING; SCOURING; CARBONISING; BLEACHING****Radioactive Soap as a Means of Studying the Rinsing of Soap from Wool**

M. Robinet and J. Roussel

*J. Textile Inst.*, 51 (Dec 1960) T 1147-T 1161

By measurements of radioactivity following the use of a soap containing  $^{14}\text{C}$  and  $^{24}\text{Na}$ , it is shown that wool washed with soap at low concentration (0.5 g/l.) retains more fatty matter after rinsing than when washed with more concentrated (3–5 g/l.) solutions, but retention of Na increases with increasing concentration of the soap. Fatty acid is deposited as a surface layer which exceeds a monomolecular film, but sodium penetrates more deeply into the fibre. The addition of sodium carbonate causes salting-out of the soap, and so gives increased retention. The use of ammonia favours the elimination of soap.

W.K.R.

**Peptidyl Shift During Wool Carbonising**

E. Hille and H. Zahn

*J. Textile Inst.*, 51 (Dec 1960) T 1162-T 1167

Chemical data are recorded for samples of commercially carbonised wool taken at various stages of production, and on some of the baked wool which had been treated with buffer solutions of various pH values under laboratory conditions. Carbonisation causes a large increase in the number of serine and threonine amino end-groups, other amino-acid end-groups remaining substantially unchanged. Neutralisation of the baked wool to pH 8.6 effects a recovery of 80% of the serine peptide bonds and of 70% in the threonine. It is recommended that carbonised wool be brought to a slightly alkaline condition (pH 8.0) as such a product is superior to partly neutralised wool.

W.K.R.

**Formation of Sulphur-containing Compounds in Wool during Carbonisation**

N. La France, K. Ziegler, and H. Zahn

*J. Textile Inst.*, 51 (Dec 1960) T 1168-T 1171

The apparent increase in the cysteic acid content of carbonised unbleached wool found by the method of low-voltage paper electrophoresis is erroneous, because two other compounds—O-serine acid sulphate and O-threonine acid sulphate—are present in the same zone. These have been shown to be partially stable to acid hydrolysis.

W.K.R.

**PATENT****Chlorite Bleaching**FBy *BP 855,281* (Germany 5 July 1957)

The goods are impregnated with a weakly alkaline or neutral aqueous solution containing a chlorite and a small amount of an amide, carbodi-imide, amino acid, amino-sulphonic acid or sulphamic acid, and are then steamed. This quickly yields an excellent bleach at 70–80°C with little evolution of chlorine dioxide gas.

C.O.C.

**Surface-active Compounds (III p. 219)**

**Nuclear Magnetic Resonance of Water Sorbed in Fibrous Materials (VI p. 225)**  
 Control of Moisture Content during Open-width Drying of Fabrics (X p. 231)

**VIII—DYEING****Studies in Adsorption****XII—Mechanism of Adsorption of Aromatic Hydrocarbons by Chromatographic Alumina**

C. H. Giles and R. B. McKay *J.C.S.*, (Jan 1961) 58–63  
 Naphthalene is adsorbed by chromatographic alumina from 2,2,4-trimethylpentane; 2,3-benzanthracene, phenanthrene, pyrene, but not naphthalene, are adsorbed from xylene. Only ca. 0.1% of the available surface is covered; S-shaped isotherms are obtained. It is suggested that a  $\pi$ -electron complex is formed between the hydrocarbon and a few sites, probably Al atoms, exposed by mechanical damage, on the outer surface of the oxide.

**XIII—Anomalous (Endothermic) Effects of Adsorption on Inorganic Solids**

C. H. Giles, J. J. Greczek, and S. N. Nakhwa

*J.C.S.*, (Jan 1961) 93–95

Adsorption of Janus Red B (C.I. 26115) and Lissamine Green BN (C.I. 44090) from water on to inorg. surfaces is apparently endothermic, saturation adsorption rising with temp.; both dyes are associated in aq. soln. From a dissociating solvent (methanol) adsorption is normal, the amount sorbed falling with rise in temp. The anomaly of adsorption from water is ascribed to partial dissociation at the substrate surface. Adsorption of dye as micelles appears to occur even from methanol, and may be due to association at the moment of adsorption. H.H.H.

**Adsorption at Inorganic Surfaces. V—Adsorption of Sulphonated Dyes by the Anodic Film on Aluminium**

C. H. Giles, H. V. Mehta, S. M. K. Rahman, and C. E. Stewart

*J. Appl. Chem.*, 9 (Sept 1959) 457–466

Quantitative adsorption tests with aromatic solutes (mainly sulphonated dyes) and chromic acid anodic alumina film are described. The adsorption mechanism of the non-chelating dyes is probably covalent bond formation (each sulphonate group replacing a hydroxy group or a chromate group attached to aluminium), together with some ion-exchange adsorption of anionic micelles. Chelating dyes form an aluminium chelate complex with the film. The sequence of events in adsorption from water appears to be—(a) film etching by rapid dissolution (< 5 min) of a surface layer of alumina, (b) rapid adsorption of an external layer of solute, (c) slow inward diffusion of solute from the external layer to complete a monolayer, (d) (with sulphonates) penetration (and ultimate breakdown) of the oxide crystal structure, (e) slow subsequent “sealing” of the film pores by growth of boehmite crystals. Process (a) influences the available surface area, which increases proportionately (and very considerably) with the volume of liquid used for adsorption. The apparent activation energy of diffusion in the film falls with increase in size of the unsulphonated residue of a dye.

C.H.G.

**Fundamental Studies on Dyeing with Disperse Dyes. II—Interaction between 1,4-Diaminoanthraquinone (DAA) and Organic Solvents**Y. Suda *J. Soc. Textile Cellulose Ind. Japan*, 16, (Nov 1960) 962–968

The solubility of DAA in a series of straight-chain alcohols and in mixtures of ethyl alcohol ( $\text{C}_2\text{H}_5\text{OH}$ ) and carbon tetrachloride ( $\text{CCl}_4$ ) was measured at different temperatures. The solubility of DAA in the  $\text{C}_2\text{H}_5\text{OH}/\text{CCl}_4$  mixture increases appreciably with increase in the proportion of  $\text{C}_2\text{H}_5\text{OH}$  and this is attributed to its greater affinity for  $\text{C}_2\text{H}_5\text{OH}$  (ca. 2.3 kcal/mol). The solubility in a series of n-alcohols indicates the existence of both polar and non-polar forces. The plot of  $\Delta H_f$  against  $\Delta S_f$  for this series of alcohols is a straight line and the point for  $\text{CCl}_4$  deviates from it. This suggests that the proportion of non-polar force to polar force in  $\text{CCl}_4$  is different from that in alcohols.

W.P.M.

**Carrier Dyeing**

H. M. Friedman

*Amer. Dyestuff Rep.*, 49 (12 Dec 1960) 946–951, 964

The comparative carrier action in the dyeing of various

polyester and cellulose triacetate fibres with different types of commercial dye carriers is discussed, and compared with the use of "mill-made" blends of an emulsifier base and the carrier chemicals—*o*-phenylenphenol, diphenyl, methyl salicylate, butyl benzoate, trichlorobenzene, and *o*-dichlorobenzene. It is shown that savings of approx. £80 per 1000 lb of commercial carrier can be made by using optimum "mill-made" blends of emulsifier base and carrier chemical, with equal carrier chemical concentration in the dyebath.

S.B.D.

#### Lightfastness of Dyes on Textiles

C. H. A. Schmitt

*Amer. Dyestuff Rep.*, 49 (12 Dec 1960) P 974—P 980

Any condition known to lower the lightfastness of goods before or during dyeing must be avoided if possible or remedied at a later stage. Among the points discussed are the cumulative effect of processing the goods, the effect of sequestering agents on copper-containing direct dyes, the optimum dyeing conditions for metal-complex and metachrome dyes, and the effect of carriers on disperse dyes on synthetic fibres. Having achieved optimum lightfastness, it is important that there is agreement between buyer and supplier as to the standards for fastness and agreement on the exact method of conducting the tests. Factors which contribute to good and bad results in both daylight and Fade-Ometer testing are reviewed.

S.B.D.

#### Migration of Aqueous Dispersions of Vat Dyes During Drying

M. R. Fox and J. F. Mawson

*SVF Fachorgan*, 15 (Nov 1960) 783—791

The authors point out that unsuitable conditions of drying can result in dye migration, producing colour differences between the face and reverse sides of the fabric. Material impregnated with a dispersion of unreduced vat dye (conditions of very low substantivity) is particularly susceptible to dye migration, if the application of heat is uneven, i.e. there is a temp. gradient across the fabric. Dye dispersion (liquid phase) migrates towards the source of greater heat, water evaporates there leaving the solid dye molecules behind. The apparatus used in the investigation was based on this principle. It consisted of a modified hair dryer, blowing hot air at 90°C, directed perpendicularly at a test fabric placed under tension at a fixed distance. Colour reflection measurements were carried out on both sides of the test fabric, and from the Kubelka-Munk equation the respective dye concentrations were calculated and were subsequently used as criteria of dye migration. Although inorganic electrolytes are of little importance as migration inhibitors on account of their detrimental effect on emulsion stability, certain thickeners and organic polyelectrolytes, e.g. sodium alginate, inhibit migration. The solution to the problem of dye migration can only be achieved by co-operation between dye manufacturers, dye users, fabric manufacturers, and machine builders.

L.A.T.

#### Drying and Migration Problems in the Finishing of Cotton

M. Peter and P. Ulrich

*SVF Fachorgan*, 15 (Nov 1960) 777—782

The authors discuss problems of dye migration during intermediate drying of cotton fabrics dyed by continuous methods. Factors which influence dye migration include—(a) construction and thickness of the fabric, (b) type of dye (e.g. Class A direct dyes are more susceptible to migration than those of Classes B and C, vat dyes of very fine particle size migrate more readily than those of coarser quality), (c) the greater the moisture pick-up during padding the greater the tendency for dye migration, (d) rapid drying and one-sided drying promote dye migration. Certain additions to the padding liquors, e.g. Stabiliser VP (Ciba), inhibit dye migration.

L.A.T.

#### Solvent-assisted Dyeing of Wool

E. M. Kärholm

*J. Textile Inst.*, 51 (Dec 1960) T 1323—T 1341

Investigations have been made of the influence of various factors on the rate of dyeing of wool with Bromphenol Blue in presence of alcohols or amines. Absorption of solvent on wool is necessary for obtaining an increase in rate of dyeing. For benzyl alcohol, the rate of dyeing is proportional to the amount of dye absorbed. By applying an acid-indicating indicator dye, it has been possible to show that

increase in rate of dyeing of the acid form can be obtained with alcohols, while increase in rate of dyeing of the basic form is obtained only with amines. Partition ratios of the two forms of indicator between buffer soln. and alcohols or amines at high liquor ratios (200:1 to 400:1) have been determined and related to dyeing curves, resulting in a hypothesis for the mechanism of solvent-assisted dyeing of wool. Rates of dyeing of surface-treated wool in presence of solvents have also been investigated. S.B.D.

#### Adsorption of Non-ionic Dyes by Wool

C. L. Bird and J. M. Firth

*J. Textile Inst.*, 51 (Dec 1960) T 1342—T 1350

Linear isotherms have been obtained on wool at 40, 60, and 80°C with derivatives of azobenzene. In most cases, with truly non-ionic dyes, the plot of  $-\Delta\mu^{\circ}/RT$  against  $1/T$  also appears to be linear, indicating constant heat of dyeing. With the weakly acidic 4-carboxy- and 4-sulphonamido-azobenzene and the weakly basic 2,4-diaminoazobenzene, heats of dyeing are not constant, probably owing to variation in ionisation at different temperatures. The most soluble of the truly non-ionic dyes examined also showed variable heats of dyeing. Non-ionic dyes have low affinity for wool and, consequently, low fibre saturation values.

S.B.D.

#### Dyeing Wool and Wool Unions with Reactive (Procion) Dyes

H. R. Hadfield and D. R. Lemm

*J. Textile Inst.*, 51 (Dec 1960) T 1351—T 1370

Effects of dyebath pH on hydrolysis, rate of reaction, and rate of exhaustion of Procion dyes on wool are discussed and information is presented on the effect of fixation on fastness properties. The relation of these properties to the practical dyeing behaviour is discussed and optimum dyebath conditions are suggested. Application from dyebaths containing solvents, both by batch-wise and by continuous methods, is outlined. Production of fast dyeings on cellulosic portions of wool-cellulosic unions with Procion dyes is described.

S.B.D.

#### Wool Surface Properties and Their Influence on Dye Uptake—A Microscopical Study

K. Köpke and B. Nilsson

*J. Textile Inst.*, 51 (Dec 1960) T 1398—T 1413

A microscopical technique was employed to localise and identify variations in the rate of dyeing between different wool fibres as well as between different parts of the same fibre. Dyed fibres were examined visually and in cross section. The causes of the variations were investigated by modifying the surface of wool fibres by chemical and/or physical means. Several dyes were used but 1:1 and 2:1 metal-complex dyes proved most useful in detecting fibre differences and these were employed for the most part. A 1:1 metal-complex dye accentuated differences both between fibres and between different parts of a fibre and was very sensitive to alkali treatment of the fibre. A 2:1 metal-complex dye revealed considerable differences between fibres and was sensitive to mechanical damage, but largely insensitive to alkali treatment. Two retarding mechanisms influencing the rate of dyeing are considered; both are surface barriers, one of an apparently mechanical kind and one of a chemical nature. An elucidation of the chemical barrier was attempted by using different chemical treatments.

W.P.M.

#### Vinylsulphone Reactive Dyes on Wool

F. Osterloh *Melliand Textilber.*, 41 (Dec 1960) 1533—1537

In neutral or alkaline soln. the vinylsulphone group of the Remalan dyes forms homopolar bonds with the amino or hydroxyl groups of wool. In acid soln. these dyes behave like ordinary acid dyes and form salt-like linkages with wool. Since the homopolar bonds are much stronger than the salt linkages, migration and levelling properties are better in acid soln. but the wet fastness is poorer. Wool dyed with Remalan dyes containing two reactive groups has a much reduced solubility in urea-bisulphite soln., indicating cross-linkage of the chains.

W.M.

#### Investigations into the Continuous Dyeing of Wool

B. Milligan

J.S.D.C., 77 (Mar 1961) 106—112

#### New Observations on the Tippy Dyeing of Wool

H. R. Hadfield and D. R. Lemm

J.S.D.C., 77 (Mar 1961) 97—106

**Dyeing and Finishing Blends of Wool and Dynel**

F. C. Rippner

*Amer. Dyestuff Rep.*, 49 (14 Nov 1960) P 856—P 857

A description of a successful method of dyeing wool-Dynel blends with mixtures of milling acid and cationic dyes in a single bath. In addition to the dyes, the bath contains Tinegal RWI, Peregal TW, Cindy AC-9, and Duponol G. Neutral-dyeing premetallised dyes may be used with an addition of diammmonium phosphate. A.J.

**Interaction of Reactive Dyes with  $\alpha$ -Amino Acids**

B. M. Bogoslovskii, A. D. Virnik, and M. A. Chekalina

*Tekhnol. Legkoi Prom.*, (4) (1960) 80—87*Chem. Abs.*, 55 (9 Jan 1961) 908

Five-fold excesses of  $\alpha$ -amino acids or their derivatives were treated in aq. alcohol with dyes containing different reactive groups. The reacting mass was refluxed for 5 h on a water bath. The products were separated and identified by paper chromatography. The  $R_f$  of the original dyes, of their hydrolysis products, and of the products obtained during the reaction of the dyes with the  $\alpha$ -amino acids were measured on the ascending chromatogram. Interaction of reactive dyes with the Na salt of glycine was studied; the amount of hydrolysed dye was 5–10% of the original dye. Under analogous conditions, acylated amino groups of  $\alpha$ -amino acids (acetylglycine), the OH group in the aliphatic chain (*N*-benzoylserine), the guanidine group of arginine (*N*<sup>2</sup>-acetylarginine), and the imino group of tryptophan (*N*-acetyltryptophan) do not react with the reactive dyes, but the dyes themselves are completely hydrolysed. Dyes do react with the imino groups of histidine, the amount of hydrolysed dye being 5–10% of the original dye. Introduction of excess of NaHCO<sub>3</sub> into the reacting mass gave no increase in the amount of hydrolysis, showing that the imino group of histidine is less active than the  $\alpha$ -amino group. Experimental methods for glycine bicarbonate,  $\alpha$ -alanine bicarbonate and *N*<sup>2</sup>-acetylhistidine bicarbonate reactions with reactive dyes are described. Data given include C and N percentages and ratios of atoms, both theoretical and experimental. 29 References. C.O.C.

**Recent Developments in the Dyeing and Printing of Dynel Synthetic Fibre**

D. J. McLeod

*Canadian Textile J.*, 77 (23 Dec 1960) 47—54

Dynel may be dyed with disperse, cationic or pre-metallised dyes at the boil; but the fibre is delustrated and softened in hot water the maximum effect occurring at the boil. If cooling from the dyeing temperature is too rapid loose stock forms a cake which is very difficult to open, and the fibre must be re-lustrated, after dyeing, by heating in the dry state to 250°F. Softening and delustring can be minimised by dyeing at 180°F using butyl benzoate as carrier. Dyeing recipes normally used at the boil need only minor adjustment for this method. Typical procedures for dyeing loose stock at the boil, without carrier and at 180°F using butyl benzoate, are given. Pile fabrics containing Dynel can be printed with selected disperse, cationic and neutral premetallised dyes. Propylene carbonate or ethylene carbonate promotes penetration of dye into the fibre and fixation is accomplished by heating, preferably in steam at 5 lb/in<sup>2</sup> (227°F), in an autoclave. Two recipes and procedures are outlined.

W.P.M.

**Dyeing and Printing Polyester and Polyester-Cellulosic Fabrics with Polyestren Dyes**

H. Musshoff

*J.S.D.O.*, 77 (Mar 1961) 89—96**Dyeing and Finishing Polyester Fibres and their Unions with Cellulosic Fibres with particular reference to the Thermosol Process**J. Müller *SVF Fachorgan*, 15 (Dec 1960) 836—848

Describes the choice and fastness properties of selected vat Indanthren dyes and speciality vat Polyestren (Cassella) dyes when applied by the Thermosol process to polyester fibres and polyester-cellulosic fibre mixtures. The material is padded with the dispersion of the dye of the right viscosity, and after intermediate drying it is subjected to a high-temp. treatment on a stenter at 210—215°C for 40—50 s. Reductive treatment which follows is carried out either in a jigger or in a chemical padder, followed by steaming and open-width drying. Solid shades and two-tone effects are possible, and application of synthetic

resins to modify the handle of the fabric presents no problems. The prospects for combined dyeing and resin application are promising.

L.A.T.

**Dyeing Polyvinyl-n-butylboronate with Congo Red**

S. Kato and Y. Tsuzuki

*Bull. Chem. Soc. Japan*, 33 (Dec 1960) 1741

A series of polyvinyl-n-butylboronates of varying degrees of esterification have been prepared, films of which have been dyed with Congo Red. The dyeings were found to be deeper than those obtained with polyvinyl alcohol itself, despite the greater hydrophobic character of the boronic esters. The amount of dye taken up increases with degree of esterification, as does the apparent diffusion coefficient. It is considered that the increased dye uptake is due to the presence of boron, a co-ordinate bond being formed between the boron atoms and the amino groups of the Congo Red molecule. The introduction of boron also makes the dense crystalline structure of polyvinyl alcohol more open, and results in an increase in the amorphous content.

I.G.L.

**Dyeing Elastic Narrow Fabrics**

M. Schmitzler

*Melliand Textilber.*, 41 (Dec 1960) 1546—1548

Three methods can be used—(1) Padding-drying, in which a certain fixation of dye occurs during drying. Only light shades of limited fastness can be obtained; (2) Padding-steaming or treating in salt soln. or combining both fixation processes. This yields deep shades of good fastness; (3) Winding on suitable holders and dyeing in package dyeing machine. This is applicable only to thin, loosely woven ribbons. Direct dyes selected for good substantivity, solubility, and fastness are mostly used. The recently introduced soluble sulphur dyes (Hydrosols) and azoic dyes can also be applied.

W.M.

**PATENTS****Dyeing Cellulosic Textiles with Vat Dyes**Standfast Dyers & Printers *BP* 856,916 (8 Apr 1958)

The material is treated with an aqueous suspension of an acid leuco vat dye [obtained by use of thiourea dioxide (C.I. Reducing Agent 11) under hot neutral or acid conditions] and then with an alkaline liquor. This yields well-penetrated full dyeings, particularly in a molten-metal dyeing machine.

C.O.C.

**Dyeing Mixtures of Cellulosic and Polyester Fibres with Vat Dyes**

HH

*BP* 856,846 (13 May 1958)

An aqueous dispersion of the dye is reduced with thiourea dioxide (C.I. Reducing Agent 11) in amount exceeding that necessary to reduce the dye under acid conditions, thus leaving some of the thiourea dioxide available to convert the dye, in presence of alkali, into the alkaline leuco derivative. The leuco vat acid suspension so obtained is applied to the textile, which is then dried, heated, impregnated with alkali, and again heated. The suspension is very stable and can be kept for several days without being re-oxidised. Its use enables uniform dyeings to be produced on mixtures of cellulosic and polyester fibres.

C.O.C.

**Dyeing or Printing Keratinous or Regenerated Protein Fibres**L. Peters and C. B. Stevens *BP* 856,381 (4 Apr 1956)

The fibres are treated with an aqueous solution or dispersion of a colorant in presence of an organic solvent for the colorant. This solvent must be liquid under the conditions of dyeing, insoluble or only slightly soluble in water, and must be a better solvent than water for the colorant. Thus wool slubbing (100) immersed in an aqueous solution (2000) containing C.I. Acid Black 600 (1), formic acid (1), Alphanol (10), and isopropyl alcohol (1) at 70°C exhausted the solution in 15 min to yield, after being kept in the bath for a further 15 min at 40°C, a well-penetrated, level dyeing, fast to scouring. In another example addition of benzyl alcohol to an acid-dye printing paste resulted in a better colour yield.

C.O.C.

**Reserve- and Cross-dyeing of Wool-Polyacrylonitrile Fibre Blends**Chemstrand Corp. *BP* 857,270 (U.S.A. 21 May 1958)

Blends of wool and acrylic (< 80% acrylonitrile, < 2% vinyl pyridine) fibres are dyed with reservation of the wool in a bath containing an acid and a 1:1 or 1:2 metal-complex dye of poor wet fastness on wool and, on the wt.

of the blend, 6–10%  $H_2SO_4$ , 0.25–2.5% of an ethylene oxide condensate with a higher fatty acid or alcohol and 0.1–2.0% of the reaction product of ethylene chlorohydrin with the condensate of 1 mol. hydroxyethyl-ethylene diamine and 1 mol. stearic acid. The dyed blend is scoured at pH 7–8 with an ethylene oxide condensate with a fatty alcohol or acid or alkyl phenol and finally cleared with a metallic formaldehyde sulphoxylate. The wool can then be cross-dyed by use of a 1:2 metal-complex dye whose dyeings on wool have good wet fastness.

C.O.C.

#### Dyeing and Printing Olefin Polymers and Copolymers

FH

BP 862,233 (Germany 11 Apr 1957)

The polyolefin is heated with a solvent dye dissolved in a chlorinated hydrocarbon resin, e.g. chlorodiphenyl resin, which is solid at room temperature, softens when hot, and when molten swells the polyolefin and penetrates into it. The dyed resin may be dissolved in a suitable solvent before it is mixed with the polyolefin.

C.O.C.

#### Coloured Glass Fibres

L.O.F. Glass Fibers Co. USP 2,932,587 (14 Sept 1956)

Water-soluble Prussian Blue (C.I. Pigment Blue 27) is used in conjunction with a suitable resin to yield a blue or bluish green, the resin and the pigment blending to yield an insoluble product, e.g. a web of glass fibres is sprayed with a solution containing Prussian Blue, a phenol-formaldehyde precondensate, and oxalic acid (to prevent precipitation), dried, and baked. The phenol-formaldehyde condensate is itself yellow, and by varying the concentration of pigment clear blues to bluish greens can be obtained.

C.O.C.

#### Hydrazine and Hydrazine Hydrate as Reducing Agents for Sulphur Dyes (III p. 220)

#### A Generalised Theory for Combination of Wool Keratin With Acid or Alkali (VI p. 225)

#### Crimping Synthetic Polymer Yarns with or without Simultaneous Dyeing (X p. 232)

### IX—PRINTING

#### Factors Affecting the Suitability of Print Pastes for Blotch Printing

Y. Okaya and J. Sanada

J. Soc. Textile Cellulose Ind. Japan, 16 (Nov. 1960) 968–975

The suitability of various thickeners for producing blotch prints with vat dyes was assessed by measuring the viscosity and spinability of the print pastes containing different amounts of thickener, and the definition, tintorial value, dye fixation, and levelness of the prints. Sodium alginate and Schiss are suitable, but a single thickener which excels from every point of view does not exist. A mixed thickener of wheat starch and sodium alginate gives excellent prints.

W.P.M.

#### Colour Terms in the Printing Trade—DIN 16 515, part 1

Die Farbe, 9 (Sept 1960) 113–124

A compilation of colour terms, some of a general nature and others specific to the printing trade, as well as definitions of terms used to illustrate the properties of printing colours and defects in practical printing. These are provisional and subject to scrutiny.

E.COATES

#### Colour Terms in Reproduction Technology and Photography—DIN 16 515, part 2

Die Farbe, 9 (Sept 1960) 125–137

Further definitions of relevant terms.

E.COATES

PATENTS

#### Printing Rollers for Producing Multi-striped Patterns on Fabrics

Établissements Victor Simon

BP 857,838 (France 26 Apr 1958)

The roller is formed of a close succession of discs of a soluble plastified colorant carried on a shaft of relatively small diameter. This enables very fine stripes to be obtained with a long service life of the roller.

C.O.C.

#### Printing on Cellulose Acetate or Hydrophilic Synthetic-polymer Fibres

FH BP 858,054 (Germany 16 Feb 1958)

When printing disperse, basic, direct, acid, vat, and solubilised vat dyes on hydrophilic fibres, deeper and more

level prints are obtained if the paste contains an oxyethylatation product obtained by reaction of 1 mol. of a naphthol, a hydrogenation product thereof, or a phenol with 2–4 mol. of ethylene oxide. This enables many dyes of the above classes which hitherto have been unsuitable for printing on cellulose acetate to be satisfactorily used for this purpose. The effect of these agents is especially marked in printing on blends of hydrophobic synthetic polymer fibres and wool.

C.O.C.

#### Printing with Synthetic-polymer Pastes

Dornbusch &amp; Co. BP 857,110 (Germany 10 Feb 1958)

The paste is supplied to a patterned impression roller so as to form a patterned layer. This layer is then transferred to the base material, e.g. cloth or paper. The container for the paste is kept at a temperature at which the paste is plastic. The impression roller is kept at a temperature which causes the paste immediately touching the roller to set while the upper portion of the paste remains plastic. The base material is kept at a temperature which causes the paste to set immediately they come into contact.

C.O.C.

#### Colouring of Photographs on Aluminium

Metaphoto Corp. USP 2,931,725 (25 Mar 1957)

Photographic silver images on anodised aluminium are heated to  $> 600^{\circ}\text{F}$  in a darkened furnace. This causes the silver image to disappear but it can be made to reappear by exposing the bleached-out, image-containing plate to ultraviolet radiation or to certain chemicals. Thus treatment with boiling 5% aq. ammonium sulphide yields brown, golden yellow, and pale bronzy yellow images on plates which have been heated to 700, 900, and  $1100^{\circ}\text{F}$  respectively. Similarly, a bleached plate treated with  $\text{Na}_2\text{S}$  and then with gold chloride yields a bright red image. Images of any colour can be obtained by suitable choice of chemicals. The images are unaffected by prolonged exposure to sunlight, highly humid atmospheres, and temperatures of 32–750°F.

C.O.C.

#### Copy Fluid for Production of Hectographic Copies from Photomechanically Prepared Master Sheets

W. Ritterfeld and G. Ritterfeld

BP 860,218 (Germany 17 Jan 1958)

A non-crystalline, hygroscopic, non-volatile oxycarbon acid dissolved in a mixture of alcohol and water is used, e.g. glycolic acid in aqueous ethanol. Its use prevents clogging of the felt with consequent stoppage of the supply of copy fluid, and also stops the master and copy-sheets from becoming too wet.

C.O.C.

#### Transfers

Dennison Manufacturing Co. BP 862,360 (12 Nov 1959)

A backing has an opaque, transferable, printed label applied to it. Additional markings are then made on the label by using a non-aqueous solution of a water-insoluble dye. Those markings diffuse through the label and are visible after reversal transfer.

C.O.C.

#### Forming Coloured Transfer Images

International Polaroid Corp.

BP 858,673 (U.S.A. 20 July 1956)

A photosensitive element including silver halide emulsion and a colorant is exposed and developed to yield an image of non-immobilised colorant at least some of which is transferred to a superposed image-receiving material. The colorant must be in a form in which it is dichroic.

C.O.C.

#### Colour Photography

Kodak BP 859,632 (U.S.A. 8 Mar 1957)

An exposed silver halide emulsion layer is treated with a solution containing a primary aromatic amino colour developer, a colour coupler, and a polyethylene glycol or derivative thereof of mol. wt.  $< 600$ . This enables cyan development to be shortened and so results in less cyan fog.

C.O.C.

#### Diffusion-transfer Reversal Process of Colour Photography

International Polaroid Corp.

BP 860,233–4 (U.S.A. 18 Feb 1957)

A process in which formation of the imagewise distribution of a colorant is controlled by alkali.

C.O.C.

#### Colour Photography

Agfa BP 861,138 (Germany 18 Sept 1956)

In colour development, presence of a compound which forms a colourless product by coupling with the oxidation

products of the colour developer makes possible substantial improvement in colour reproduction, especially with multi-layer materials. Examples of such compounds are  $\omega$ -cyanacetophenone and 4-diphenyl-5-pyrazolone.

C.O.C.

Colour Embossing Calenders (I p. 218)

Colour Couplers (IV p. 222)

Dicarbocyanine Dyes (IV p. 223)

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Recent Developments in the Dyeing and Printing of Dynol Synthetic Fibre (VIII p. 229)

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Dyeing and Printing Olefin Polymers and Copolymers (VIII p. 230)

## X—SIZING AND FINISHING

### Control of Moisture Content during Open-width Drying of Fabrics

R. Burgholz *SVF Fachorgan*, 15 (Nov 1960) 718-740

Deals with aspects of uniformity and quality of drying in relation to energy input and running costs of drying. Generally speaking, the time of drying is inversely proportional to the square of temp. Hence the temp. must be adjusted if the running speed of the fabric is varied. To avoid damage through overdrying, the moisture content in the fabric and in the drying medium must be controlled. The main methods of measuring residual moisture in the fabric are based on changes in the electrostatic, dielectric, and resistance properties of the material. The author discusses in detail various systems of measuring and controlling residual moisture in the fabric during drying, using hot air, superheated steam, or hot air-steam mixture as drying media. 17 References. L.A.T.

### Modification of Cotton with Formaldehyde

L. H. Chance, R. M. Perkins, and W. A. Reeves

*Text. Research J.*, 31 (Jan 1961) 71

A method which has given good results on pilot plant is to treat cotton fabric with an aqueous solution of formaldehyde, hydrochloric acid, and acetic acid at room temperature. Solutions containing 9-18% water gave optimum high wet and dry crease recovery. C.O.C.

### Polymerisation of Ethylene Ureas and Ethylene Amides within Cotton Fibres

F. B. Jones, H. G. Hammon, R. I. Leininger, and R. C. Heiligmamn *Text. Research J.*, 31 (Jan 1961) 57-65

Of several bisethylene urea, bisethylene amide, and bisethylene urethane compounds tested for their effectiveness as reactive finishes for improving the crease resistance of cotton cloth, the diaziridinyl derivatives of hexamethylene di-isocyanate and mono- and di-ethylene glycol bis(chloroformate) are capable of imparting considerable improvement to crease resistance as measured by crease-recovery angle. These compounds can be applied as aqueous solutions or dispersions by conventional padding methods. On curing they polymerise with and probably around the fibrils of the fibres. Cloth treated with the diaziridinyl derivative of hexamethylene di-isocyanate is particularly resistant to loss of crease resistance after repeated laundering. However, as applied, the bisethylene amides, ureas, and urethanes used were susceptible to retention of chlorine from bleach liquors. C.O.C.

### Studies on Textile Finishing Agents. XIX—Polymerisation and Condensation Reactions of *N*-methylolacrylamide within the Fibre

H. Kamogawa, R. Murase, and Y. Sekiya

*J. Soc. Textile Cellulose Ind. Japan*, 16 (Nov 1960) 980-987

The changes of physical properties of spun viscose rayon and cotton with inner polymerisation and condensation reactions of *N*-methylolacrylamide (MAM) and acrylamide (AM) were investigated. Although some cross-linking between polymer chains was observed when AM was polymerised in cotton the crease recovery and strength were not much improved. Treatment of viscose rayon with MAM gave excellent crease- and shrink-resistant finishes of

excellent wash fastness. AM-treated fabrics had excellent abrasion resistance. The poor wash fastness and shrinkage on washing of dimethylolurea-finished fabrics can be improved by admixture of MAM. Aftertreatment of MAM-treated fabrics with hexamethylenediamine and formaldehyde gives excellent crease recovery and some improvement in strength. Cellulosic and cellulosic union fabrics so treated can be dyed with acid dyes. W.P.M.

### Chemical Mechanisms in Chlorine Retention by Resin-treated Cotton Fabrics

AATCC Piedmont Section

*Amer. Dyestuff Rep.*, 49 (14 Nov 1960) 843-855

Chlorine is retained by resin-treated fabrics by the formation of chloroamides by free NH groups in the resin. Simultaneously, oxidative reactions can occur which also involve free NH groups, and which lead to degradation of the resin molecule. Resin chloroamides are subject to further decomposition, particularly at high temperature, leading to degradation of the cellulose. Degradation occurs by liberation of atomic chlorine from the chloroamide, which then extracts hydrogen from the cellulose to form hydrogen chloride. Degradation of the cellulose is due primarily to attack by HCl rather than by Cl. Degradation can be reduced by the use of inhibitors which react with Cl and by the presence of alkaline materials—which may be the resin itself—to neutralise HCl. A.J.

### Permanent Setting of Wool

A. J. Farnworth

*Amer. Dyestuff Rep.*, 49 (26 Dec 1960) P 996-P 1001

Evidence is summarised for the conclusion that permanent set in wool fibres results from the rearrangement of hydrogen bonds following the breaking of some disulphide bonds in the peptide chains in wool. Permanent set may be achieved under certain conditions in a relatively short time and this led to the development of the Si-Ro-Set process using ammonium thioglycolate which is now used on a commercial scale in garment pressing to obtain permanent creases. One of the major problems encountered was the alteration in shade by the reduction of dyes. Tables have now been published by dye manufacturers giving the fastness figures for wool dyes to the Si-Ro-Set process. "Non-iron" fabrics may be produced by first treating the fabric with  $KMnO_4$  in concentrated salt solution followed by a clearing treatment with sodium bisulphite; this gives a high degree of shrinkage resistance. On subsequent treatment by means of the Si-Ro-Set process a washable "non-iron" effect is obtained. It was found, however, that on an industrial scale sodium bisulphite was more suitable for application than ammonium thioglycolate. Results of other finishing treatments and tests made on treated fabrics are given.

P.G.M.

### Dimensional Stabilisation of Wool Fabric by Interfacial Polymerisation

R. E. Whitfield, L. A. Miller, and W. L. Wasley

*Text. Research J.*, 31 (Jan 1961) 74

Formation of polyamides, e.g. polyhexamethylene sebacamide, on the surface of the fibres by interfacial polymerisation when the cloth is padded consecutively through an aqueous solution of a diamine and a water-immiscible solution of a diacid dichloride, renders wool fabric shrink resistant. No heating or curing is necessary. Less than 5% of polymer on the fabric gives dimensional stability in laundering. Handle, mechanical strength, and chemical resistance of the wool remain unchanged.

C.O.C.

### Development of Wash-and-wear Fabrics for Modern Home Laundering

A. E. Brown and J. F. Krasny

*J. Textile Inst.*, 51 (Dec 1960) T 1414-T 1437

The effects of yarn and fabric construction variables on the wash-and-wear performance of worsted fabrics were investigated. Of 123 fabrics studied 34 did not shrink and an additional 43 shrunk between 0 and 2% in area after 10 machine washing and drying cycles. Finer rather than coarser yarns and compact construction were most important for reduction of shrinkage and fuzz formation. Fuzz formation was also decreased by measures which reduced the raising of fibres by rubbing between yarns and between fabrics, such as the use of two-ply instead of single yarns, opposite twist in adjoining and cross-wise yarns, and similar diameters in warp and weft yarns.

Though most of the fabrics were muss resistant, muzziness increased with decreasing fabric weight, the use of coarser single rather than finer two-ply yarns, and low twist. The results indicate that fabrics of satisfactory wash-and-wear performance can be constructed from untreated wool; but chemical stabilisation of fabrics will broaden the range of suitable fabric constructions. W.P.M.

**Improvements in Epoxy-amino Polyamide Finishes for Easy-care Wool Fabrics**

C. E. Pardo. *J. Textile Inst.*, 51 (Dec 1960) p 1462—p 1474

Improved finishes for control of felting shrinkage in wool apparel fabrics, obtained with blends of glycerol-based and bisphenol A-based epoxy resins with an amino polyamide resin, are described. Oxidative pretreatment of the fabric improves the shrinkage resistance by increasing the adhesion of resin to fabric. Replacement of the glycerol-based resin with increasing proportions of bisphenol A-based resin gives increasing shrinkage protection which is accompanied by increased flexural rigidity. Much of the stiffness imparted by the resin treatment is removed by mechanical manipulation during washing after curing. The resins may be applied by padding or exhaustion techniques, though the latter tend to give uneven results. Successful application of a typical resin finish to full-size woolen fabrics by a pad, dry, cure, back-wash sequence is described. A worsted piece required a second treatment to achieve a satisfactory degree of shrinkage resistance. W.P.M.

**Washable Non-iron Fabrics from Wool**

A. J. Farnworth, M. Lipson, and J. R. McPhee

*J. Textile Inst.*, 51 (Dec 1960) p 1504—p 1516

The flat setting of shrinkproofed wool can give washable, non-iron effects on pure wool fabrics. Setting, which consists in steaming fabrics containing 50% by weight of 1% sodium bisulphite solution for 5 min on a blowing machine, prevents surface distortion of the fabrics during subsequent washing. 17 Specially constructed fabrics were made satisfactorily shrink resistant and set, and the combined treatments tended to increase wet crease recovery. However, wet crease recovery is not directly related to the non-iron property of treated fabrics. The overcoming of dyeing problems associated with the treatments, the prevention of stiffening during setting and the production of fabrics free from relaxation shrinkage are discussed. Difficulties in constructing washable, minimum-iron garments from treated fabrics are discussed and recommendations are given for precautions to be taken during making up. W.P.M.

**Silicone Finish on Synthetic-fibre Fabrics**

C. Smith

*Chemiefasern*, 12 (Dec 1960) 824—828

Results of Bundesmann tests on silicone-treated nylon, cellulose acetate, Terylene, cotton, and wool are shown graphically. If lower concn. are used a certain degree of water repellency and good resistance to staining by aqueous soln. are obtained. Silicone finish much improves the sewing properties of fibres by increasing the tearing strength and decreasing friction and abrasion, which is of special interest for modern high-speed sewing machines. New silicone preparations have recently been put on the market which do not require high temp. condensation. Another use for silicones is as softeners for wash-and-wear finishes whereby tearing strength, resistance to abrasion and crease recovery are improved at the same time. W.M.

**Finishing Fabrics of Polyester Fibres and of Unions with Wool, Cotton, or Spun Viscose**

G. Raunenberger and J. Thimm

*Chemiefasern*, 12 (Dec 1960) 819—823

Articles consisting of 100% polyester fibre (Trevira filament) require washing (non-ionic detergent and alkali), heat-setting (190°C for ca. 30 s), bleaching (chlorite), antistatic finish. Staple-fibre fabrics must be singed to reduce pilling. Polyester fibre material can be embossed (temp. 180—210°C, pressure 1—10 tons). By treatment with aqueous alkalis fibres of the Trevira type can be "peeled off", i.e. the outer layers can gradually be removed; fibres of finer denier and softer silk-like handle are obtained thereby. Polyester-wool articles are washed, crumbed, dried without tension, set at temp. of 175—185°C for 20—35 s. Fabrics are allowed to shrink by 3—8% in both directions during setting. Washing after setting improves handle and crease recovery. Steaming and brushing are

advisable followed by shearing, singeing (if required), pressing and decatising. Poplin-like articles made of Trevira-cotton mixtures are desized, washed, bleached, dyed, set, singed, mercerised, stentered, calendered. Sanforised Trevira-spun viscose articles for outerwear are desized, washed, set, dyed, dried, singed, washed, softened, stentered, and calendered, if necessary. W.M.

**PATENTS**

**Crimping Synthetic Polymer Yarns with or without Simultaneous Dyeing**

British Nylon Spinners *BP* 857,721 (7 June 1958)

When crimping synthetic-polymer, e.g. nylon, yarns by any of the usual processes, the quality of the crimp is improved if the yarn-supply package mounted on the machine is immersed in hot, preferably boiling, water or aqueous dye solution. The resulting yarn has improved loftiness, probably because its fibres are swollen before they are crimped. C.O.C.

**Impregnating Fibrous Sheets or Webs**

Witco Chemical Co. *BP* 856,389 (24 Dec 1957)

Varying degrees of impregnation of a web which may be appreciably thick are obtained by applying the impregnant as a foam to one side of the web and applying suction on the other side to suck the impregnant into the web. C.O.C.

**Temporarily Water-resistant Tissues**

Vereinigte Papierwerke Schickedanz & Co.

*BP* 857,162 (Germany 7 Feb 1957)

A non-woven fabric of natural or synthetic fibres containing hydroxyl groups is treated with a solution of a hydroxylic colloid and a compound containing an aldehyde group, polyethylenimine, or a di-isocyanate, the two components being in the proportion by weight of from 4:1 to 1:2. Suitable components are methylcellulose and melamine-formaldehyde. The products are suitable for use as disposable handkerchiefs, towels, underwear, etc. C.O.C.

**Treating Cellulosic Fabrics with Synthetic Resins**

Toot Broadhurst Lee Co. *BP* 855,509 (1 Nov 1955)

Cotton or linen fabrics are impregnated with a lubricant containing an amide group and with a 3:1 formaldehyde-melamine precondensate and then baked. The amount of lubricant is such that the finished fabric contains 9.2—3.0% of lubricant on its dry weight. Thus addition of Sapamine WP (Ciba) to a formaldehyde-melamine liquor applied to cotton poplin resulted in finished cloth having a better "minimum-iron" finish than that obtained in absence of the lubricant. C.O.C.

**Resin Finish for Cellulosic Textiles**

American Cyanamid Co. *USP* 2,929,743 (8 Nov 1956)

Good crease recovery and a soft, full, dry handle are obtained by impregnating with (1) a heat-setting aminoplast which is at least partially water-soluble, and (2) a water-insoluble thermoplastic copolymer obtained by polymerising an azeotropic mixture of a styrene and an alkyl acrylate (Alk of 1—4 C). There are 2—4 parts of the aminoplast for each part of the copolymer. The impregnated material is then baked to insolubilise the aminoplast. C.O.C.

**Drip-dry Finish**

Deering Milliken Research Corp.

*BP* 855,547 (U.S.A. 3 Apr 1956)

Cellulosic textiles are swollen and then treated with a cross-linking agent which gives a cross-link including a chain of > 2 C. Thus mercerised cotton fabric is impregnated with 15% of its weight of a 7:3 mixture of 2,3-dichloro-1-propanol and 1,3-dichloro-2-propanol, followed immediately by impregnation with 70% (on weight of fabric) of 10% NaOH. The impregnated fabric is tightly wound, wrapped in a sheet of polyethylene, and stored at room temperature for 12 h. Finally it is scoured and dried. This yields a fabric having excellent flat-drying properties, and an excellent white which does not tend to yellow when repeatedly bleached with chlorine. C.O.C.

**Water-repellent Agents**

FBy *BP* 855,803 (Germany 17 Sept 1956)

Aqueous emulsions of waxes containing chromium-complex salts, of the Werner type containing acyl groups, and cationic emulsifying agents are used. They can be used together with resinous precondensates to improve

both the fastness to washing of the water-repellent finish and the crease-shedding properties of the fabric. C.O.C.

#### Durable Flame-resistant Finish on Cellulosic Material

E. L. Donahue *BP 856,360* (U.S.A. 26 Sept 1958)

The material is treated with a urea phosphate and sufficient of a pyridinaceous substance to catalyse esterification of the cellulose by the urea phosphate. Thus cotton is impregnated with its own weight of a solution made up of urea phosphate (25.0 g), Cellosolve (7.0), pyridine complexes (bone oil) (0.2), Na alkylacyl sulphonate (0.2), and water (65.0), dried at 200°F and then baked at 324°F for 3 min. This yields a good flame-resistant finish with little effect on the handle or tensile strength of the fabric. C.O.C.

#### Flame-resisting Finish on Cellulose Acetate Fibres

Courtaulds

*BP 858,582* (20 Nov. 1956)

Bis-(2-chloroethylamino)phosphonate ( $\text{ClCH}_2\text{CH}_2\text{O}_2\text{P}(\text{O})(\text{NH}_2)_2$ ):  $\text{PO-NH}_2$  is incorporated from aqueous or organo solvent solution into the fibres at any stage of their manufacture or processing. For a satisfactory flame-resistant finish, the fibres should contain  $< 1\%$  of phosphorus. C.O.C.

#### Oil-repellent Finish

Bradford Dyers' Association *BP 858,022* (5 Nov 1957)

Fibrous materials are treated with a film-forming oleophobic fluorocarbon compound and a compound which reduces the swelling in water of hydrophilic fibres. Use of the two compounds gives improved water-repellency compared with the use of the fluorocarbon compound alone and also improves the durability of the combined oil- and water-repellent effect. Thus, application of a fluorocarbon-carboxylic acid-chromium complex and then of a polyfunctional isocyanate to textiles gives good fastness to washing without any reduction in the oil-repellent effect imparted by the fluorocarbon compound. C.O.C.

#### Compressive Shrinking

Compax Corp. *BP 855,079* (U.S.A. 4 Feb 1957)

One surface of a fabric, particularly tubular knit fabric, is brought into contact with a large surface area of a rigid feed roller with which the fabric has low frictional gripping characteristics. The fabric is held against this roller in absence of substantial local pressure up to a first line forming the start of a treatment zone. Then another surface of the fabric is pressed against a retarding roller having a peripheral speed less than that of the feed roller, there being high friction between the roller and the fabric. The fabric is subjected to localised pressure between the rollers along a line 0.25 in. from the first line, so as to compress the fabric between the lines. C.O.C.

#### Rot- and Insect-proofing

Commercial Solvents Corp.

*BP 858,811* (U.S.A. 30 July 1957)

1,3-disubstituted-5-aminohexahydropyrimidines effectively protect non-insect-produced textiles and paper against mildew, bacteria, and moths. Thus, wool impregnated with a 5% alcoholic solution of 5-amino-1,3-bis(2-ethylhexyl)-5-methylhexahydropyrimidine, squeezed, and allowed to dry for 2 weeks was immune to attack by larvae of the black carpet beetle. Cotton impregnated with a 10% solution of the same compound in acetone was unaffected by a 7 weeks' soil-burial test in which the untreated control was completely rotted after 7 days. C.O.C.

#### Acylmercaptoamides in the Permanent Setting of Hair

Société Monsavon l'Oreal

*BP 860,705* (France 17 Apr 1958)

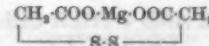
A reducing agent for use in permanent setting of hair has formula  $\text{R}^1\text{CO-S-CHR}^2\text{CO-NHR}^3$  ( $\text{R}^1 = \text{Alk}$  or carboxyalkyl;  $\text{R}^2$  and  $\text{R}^3 = \text{H}$  or  $\text{Alk}$ ), e.g. *S*-acetylmercaptoacetamide. C.O.C.

#### Magnesium Thioglycolate as Reducing Agent in Permanent Waving

Gillette Co.

*BP 858,216* (U.S.A. 9 Nov 1956)

Treating  $\text{MgCO}_3$  in water with its equivalent of thioglycolic acid yields  $\text{HS-CH}_2\text{COO-Mg-CH-COO-CH}_2\text{SH}$ , which may be isolated as a colourless solid, stable in air and readily soluble in water. On oxidising its aqueous solution—



is obtained, also as a colourless, stable solid readily soluble in water. They are excellent reducing agents for use in the permanent waving of hair, but in the case of the thioglycolate an additional reducing agent must be present to reduce it first to the thioglycolate. C.O.C.

#### Removing Glaze from Synthetic-polymer Fibres

DuP *USP 2,930,099* (12 July 1954)

Glaze impressed on fabric containing a yarn of synthetic linear polymer fibres is removed by heating the fabric in absence of tension at least to approx. the temperature at which glaze is imparted to the fabric and below m.p. Glaze removal is accompanied by increase in fabric bulk. C.O.C.

#### Grafting Monomers to Hydrogen-containing High Polymers

G. Öster *BP 856,884* (U.S.A. 21 Sept 1956)

A natural or synthetic polymer is irradiated with radiation of wavelength 170-300 m $\mu$  in presence of an organic activator for long enough to allow free radicals to form. It is simultaneously or subsequently treated with a polymer containing at least one ethylenically unsaturated monomer capable of polymerising in presence of free radicals. Thus polyacrylonitrile fabric was soaked in a 50:50 mixture of ethanol and dimethylformamide containing 2% of diphenylamine. It was then rinsed with water, immersed in aqueous 50% acrylic acid at a depth of  $\frac{1}{2}$  in. from the surface and then irradiated with a 15-W G.E.C. germicidal lamp at a distance of 3 in. for 10 min. The resultant material had strong affinity for basic dyes, whereas the untreated material had none. C.O.C.

#### Coating Nylon and Polyester Fabrics by Use of Irradiation

DuP *BP 856,329* (U.S.A. 18 Oct 1956)

Modification of *BP 801,531* (J.S.D.C., 74 (1958) 810). The substrate is irradiated with  $< 0.01$  W-s/cm $^2$  of substrate surface of ionising radiation in presence of oxygen of concentration  $10^{15} - 5 \times 10^{16}$  mol/cm $^2$  of substrate surface. It is subsequently stored, if desired, and then brought into contact with the desired monomer without access to any additional quantity of oxygen. Thus nylon and polyester fabrics were irradiated in a hermetically sealed glass vessel evacuated to an absolute pressure of 1 mm. Subsequently they were treated with methyl acrylate vapour, light and air being excluded from the fabrics throughout both processes and the intervening storage period. Both fabrics increased considerably in weight and were stiffer, tighter, and drier in handle. They were much less readily penetrated by water and so more useful for tenting and tarpaulins than the untreated fabrics. C.O.C.

#### Floor Covering

Pneumatiques & Caoutchouc Manufacture Kleber Colombes

*BP 858,910* (France 20 Feb 1957)

A base fabric is coated with a cellular thermoplastic synthetic resin and a top coating of the same resin in non-cellular form. C.O.C.

#### Multilayer Coating (I p. 218)

Colour Embossing Calenders (I p. 218)

Vapourising-coating with Metals (I p. 219)

Laminating Machine and Method (I p. 219)

Setting Properties of Keratin Fibres in Relation to Structure (VI p. 225)

Migration of Aqueous Dispersions of Vat Dyes During Drying (VIII p. 228)

Drying and Migration Problems in the Finishing of Cotton (VIII p. 228)

Dyeing and Finishing Blends of Wool and Dynel (VIII p. 229)

Dyeing and Finishing of Polyester Fibres and their Unions with Cellulosic Fibres with particular reference to the Thermosol Process (VIII p. 229)

Adhesion of some Elastomers to Fibres and Foil of Viscose Rayon Modified with Methyl and Isopropyl Isocyanates (XIII p. 235)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

### Stability of Cellulose in Cadoxen Solutions

H. Vink *Svensk Papperstidning*, 64 (31 Jan 1961) 50–52  
(in English)

Degradation of cellulose is only partly due to the presence of oxygen but is catalysed by impurities, e.g. transition metals, especially  $\text{Co}^{++}$ . In pure solutions there is little degradation and in the case of macromolecular measurements it can be ignored.

R.A.

### The Paper "Pile" Theory: a Simplified Concept of Opacity and Transparency of Paper

G. Schmidt *Das Papier*, 14 (1960) 445–452  
*Chem. Abstr.*, 55 (9 Jan 1961) 986

Opacity and transparency of paper depend upon its thickness but when comparing different types of paper this dependence should be eliminated and the optical properties of the paper so formulated that they can be expressed by a dispersion coefficient independent of thickness or basis weight. By using piles of paper containing varying numbers of sheets mathematical formulae were developed which related the light reflectivity of a single sheet to its thickness. The single sheet was then considered as a pile consisting of various layers within the sheet. Formulae so developed were compared with the Kubelka–Munk theory and checked experimentally. Such simple formulae are appropriate to practical measurements and for calculating opacity and transparency. Formulae are also derived which serve for comparative measurements of papers having different basis weights.

C.O.C.

### PATENTS

#### Esters in Rosin Sizes

P. K. Wieger *BP* 859,787 (Germany 28 May 1957)

Sizes which do not require high-temperature drying can be used with papers glazed on one side and which yield better sizing than normal rosin sizes contain as sizing agent (1) a rosin ester and/or (2) esters and/or polyesters, with polyhydric alcohols or amino alcohols, of adducts produced from rosin by the Diels–Alder reaction with a dienophylic mono- or di-carboxylic acid or anhydride, and/or (3) adducts of a dienophylic mono- or di-carboxylic acid or anhydride with a rosinic ester.

C.O.C.

#### Coating Paper with Acylated Protein

Glidden Co. *USP* 2,932,589 (12 Mar 1956)

Dilute alkaline solutions of soya protein when acylated with a dicarboxylic acid anhydride yield a product suitable for coating paper. The product can hold a high proportion of solids contents without becoming unduly viscous. Coating compositions so formed are at least as good as compositions containing high-grade casein as the adhesive.

C.O.C.

#### Drum-finished Coated Paper

S. D. Warren Co. *BP* 862,115 (8 Oct 1959)

A jet of steam impinges against the coated, wet or dry, web immediately before it contacts the drying cylinder.

R.A.

#### Carbon Paper

Columbia Ribbon & Carbon Manufacturing Co.

*USP* 2,931,752 (16 Sept 1953)

Paper, before it is finally dried on a paper-making machine if desired, is continuously coated with a composition containing wax (8 parts by wt.), fatty acid and alkali (10–20), colorant (16–32) and water (sufficient to provide coating consistency).

C.O.C.

#### Carbon Paper

Kee Lox Manufacturing Co. *BP* 860,769 (13 Apr 1959)

Paper is coated with a plastisol, a plasticiser, a colorant, and a non-plasticising non-volatile diluent colorant carrier. This composition has low viscosity at room temperature and when heated fuses to form a firm, hard, non-tacky film on the substrate.

C.O.C.

#### Pressure-sensitive Transfer Record Sheet

National Cash Register Co. *USP* 2,932,582 (4 Feb 1959)

A porous web, e.g. tissue paper, is given a first coating containing minute pressure-rupturable capsules containing a liquid which can be released by rupturing the capsules. There is a top coating of a solid which is soluble in the liquid in the capsules and which contains a colorant which is rendered sufficiently mobile by the liquid to render the top coating and colorant transferable.

C.O.C.

## Self-marking Manifolding Sheets

Columbia Ribbon & Carbon Manufacturing Co.

*BP* 858,419 (19 Feb 1957)

Paper is coated with a spongy, compressible coating containing a colorant dispersed in it so as to only lightly tint the coating until it is compacted by pressure, when the intensity of colour is much increased. An example of such a composition is ethylcellulose (9 parts by wt.), acetone (41), Microcrystalline Be square wax (4),  $\text{CCl}_4$  (50), 20% Blue lake dispersion (12), and water (20).

C.O.C.

### Multilayer Coating (I p. 218)

### Vaporising-coating with Metals (I p. 219)

### Ink for Carbon Paper (V p. 224)

### Cellulose-*p*-xylyl-bis-ether—a Yellow Compound having Green Fluorescence (VI p. 226)

## XIII—RUBBER; RESINS; PLASTICS

### Syntheses of Some Polymeric Polypeptides of Lanthionine

M. Frankel and D. Gertwer *J.C.S.*, (Jan 1961) 450–462

Polymers of lanthionine having defined structures are obtained by polymerisation of suitable protected monomers, e.g. the *N*-carboxy-anhydrides of mono-*N*-benzoyloxy-carbonyl-lanthionine monobenzyl ester and of the adjacent monomethyl ester of *N*-acetyl-lanthionine. The synthesis of *S*-2-methoxy-carbonyl-ethyl-*L*-cysteine from methyl acrylate and *L*-cysteine and its polymerisation are also described.

H.H.H.

### Transition Phenomenon of Unsaturated Polyester Resin

K. Tanaka

*Bull. Chem. Soc. Japan*, 33 (Dec 1960) 1702–1706

The polyester resins under consideration are those cross-linked by a vinyl compound, e.g. styrene or vinyl acetate. Resins of this type have two transition points of second order, the upper transition temperature being dependent on the extent of the cross-linking. Resins examined were (a) the M.D. series, based on the resin obtained from maleic anhydride and diethylene glycol in a molar ratio of 1:1, and (b) the M.A.D. series, prepared from maleic anhydride, adipic acid and diethylene glycol in molar ratio of 9:1:10. From these starting materials cross-linked resins were obtained by treatment with styrene, giving a styrene monomer content varying from 10–35% and 10–40% in the M.D. and M.A.D. series respectively. In the former series, the transition below 0°C took place at approximately the same temperature in all cases, whereas the upper transition temperature was found to rise with increasing styrene content, this being evidence for the association of the higher temperature transition with the degree of cross-linking. Similar results are reported for the M.A.D. series, although densities and transition temperatures are lower than those of the M.D. series, and there is a less marked dependence on the styrene content, a result of less dense cross-linking owing to the adipic acid content. Results of nuclear magnetic resonance absorption measurements are reported, and the origin of the two second-order transitions is discussed.

I.G.L.

### Design of Unsaturated Polyester Resins for Surface Coatings

V. F. Jenkins, A. Mott, and R. J. Wicker

*J. Oil & Col. Chem. Assoc.*, 44 (Jan 1961) 42–60

Design of unsaturated polyester systems is discussed in relation to the problems of "air-inhibition" (failure to cure properly at air interfaces) and gel-time (effective pot-life). Air-inhibition may be overcome either by building allyl ether groups into the polyester chain, or by using a low-molecular-weight polyallyl ether as the monomer; the latter method minimises loss of monomer by evaporation and is preferred. Certain unsaturated polyesters with melting points > 90°C give surface films that are tack-free; they are, however, brittle. The lower aliphatic oximes extend gel-times without appreciably increasing drying times; they are most successful in wax-free polyester systems based on monomers of low volatility.

J.W.D.

**Adhesion of some Elastomers to Fibres and Foil of Viscose Rayon Modified with Methyl and Isopropyl Isocyanates**

T. Antczak

*Zesz. Nauk. Politech. Lodz.*  
(28 Chemia 8) (1960) 41-67

Methyl and isopropyl isocyanates were prepared and stored in gaseous form. Previously dried viscose rayon fabric, cord, and foil were treated with gaseous isocyanate in an enclosed vessel in presence of a chlorinated hydrocarbon of low b.p. The isocyanate condenses with some of the secondary and primary hydroxy groups of the cellulose. As a result a hydrophobic product containing 1.10%–1.25% of nitrogen is obtained. The handle of the materials remains unchanged and the cellulose urethanes produced do not decompose into gaseous products during vulcanisation. Natural rubber and polyisobutylene compound adhere very strongly to this modified cellulose. Methyl isocyanate gives better results than isopropyl isocyanate. T.Z.W.

PATENTS

**Rubber Pigmented with Silica (C.I. Pigment White 27)**

B. F. Goodrich Co. *BP 858,742* (U.S.A. 28 Aug 1958)

A butadiene-styrene copolymer is pigmented with silica in presence of a compound of formula  $R^1-SS-NR^2R^3$  ( $R^1$  = thiazole of 3–14 C;  $R^2$  and  $R^3$  = aliphatic, cycloaliphatic or aralkyl or together form a single alicyclic or heterocyclic ring) as curing agent, e.g. 2-benzothiazyl-*N*-morpholinyl disulphide. Other pigments, fillers, etc. may be present. C.O.C.

**Decorative Plastic Floor or Wall Coverings**

Goodyear Tire & Rubber Co.

*BP 857,254* (U.S.A. 31 Mar 1958)

Machine in which granules of a thermo-flowable plastic are formed into a sheet and the spaces between them filled with a transparent plastic having different light-reflecting properties to the granules. The granules retain their individual identity throughout. C.O.C.

**Vaporising-coating with Metals (I p. 219)**

**Ion-exchange Fibres, Films, and the Like (VI p. 227)**

Grafting Monomers to Hydrogen-containing High Polymers (X p. 233)

**XIV—ANALYSIS; TESTING; APPARATUS**

**Chromatographic Behaviour of Some Stereochemically Interrelated Flavonoid Compounds in Aqueous Medium**

D. G. Roux, E. A. Maihs, and E. Paulus

*J. Chromatography*, 5 (Jan 1961) 9–15

The effects of the following factors on  $R_f$  values of enantiomorphs of some flavonoid cpds., including C.I. Natural Brown 1 and C.I. Natural Brown 3, have been evaluated—(a) separation of enantiomorphous flavan-3-ols, flavan-3,4-diols, and 2,3-dihydroflavonols; (b) *cis-trans* relationship of substituents in the heterocyclic ring of flavan-3-ols, and (c) introduction of a carbonyl group in 4-position, hydroxyl groups in 4,5, and 5'-positions, and a galloyl group in 3-position. Zero  $R_f$  value of planar flavonoid structures is attributed to low aqueous solubility. Aqueous solubility appears to be a dominating factor affecting all  $R_f$  values. F.J.

**Chromatography of Flavonols in the Solvent System Chloroform-Acetic Acid-Water**

K. Egger *J. Chromatography*, 5 (Jan 1961) 74–81

The  $R_f$  values of several flavonols were found to be dependent on composition of the title solvent system. Mixtures containing 25–70% chloroform were suitable.  $R_f$  relationships made it possible to obtain kaempferol free from troublesome impurities and to obtain good separation of methyl ethers from the corresponding hydroxyl cpds. A sharp separation of the rhamnosides, glucosides, and their mixtures from myricetin, quercetin, and kaempferol was also achieved.  $R_f$  values of 24 flavonols determined for five solvent mixtures of chloroform and acetic acid ranging in composition from 1:2 to 2:1 by vol. are given. F.J.

**Polarographic Assays of Permitted (Italy) Food Dyes**

E. Cerma *Rasse. chim. per chim. e indus.*, 12 (1) (1960) 13–20

*Chem. Abs.*, 55 (23 Jan 1961) 1953

Polarograms of the 13 food dyes permitted in Italy were

taken in a photorecording AME polarograph at  $25 \pm 1^\circ\text{C}$  (dropping Hg and standard HCl electrodes). The de-aerated solutions were kept under nitrogen during determinations. They were buffered with 0.2–0.4  $(\text{CH}_3)_3\text{NOH}$  or 0.2–0.4 NaOH, and 1% gelatin was added to eliminate interfering maxima. Tables show the pH range permitting identification, the ratio  $\Delta E_{0.5}/\Delta \text{pH}$  in this range at one or more levels,  $E_{0.5}$  extrapolated to pH 0 and the final identifying ratio  $E_{0.5}/\text{pH}$  characterising each dye. Quinoline Yellow (C.I. Food Yellow 13) and Patent Blue V (C.I. Food Blue 3) each had four such ratios; Azorubine, Ponceau 4R and 6R, and Amaranth (C.I. Food Red 3, 7, 8, and 9) three each; Brilliant Black RN, Erythrosine (C.I. Food Red 14) and Indigo (C.I. Food Blue 1) two each; Scarlet GN (C.I. Food Red 2), Fast Red E (C.I. Food Red 4), Tartrazine (C.I. Food Yellow 4) and Sunset Yellow (C.I. Food Yellow 3), one each. C.O.C.

**Food Colours**

A. Fouassin *Farmaco (Pavia) Ed. prat.*, 15 (1960) 453–470  
(in French)

*Chem. Abs.*, 55 (23 Jan 1961) 2020

A review of the dangers of use of dyes in foods, criteria of admissibility, list of accepted synthetic food dyes, and methods of identification with special regard to chromatography. 119 References. C.O.C.

**Detection of Riboflavin in Artificially Coloured Food Pastes**

F. Di Stefano and D. Renzi

*Rend. ist. super. sanità*, 19 (1956) 294–297

*Chem. Abs.*, 55 (23 Jan 1961) 1953

If Nebbia's method (*Chem. Abs.*, 46 (1952) 3172) for detecting riboflavin in food pastes long after their manufacture gives a doubtful or negative result, the detection of lumichrome formed by decomposition of riboflavin in ultraviolet radiation is proposed. The sample is extracted in the cold by centrifuging with water, exposed to ultraviolet radiation for 45 min, which facilitates formation of lumichrome, and then examined in Wood's light. A blue fluorescence, characteristic of lumichrome, indicates that the paste was artificially coloured with riboflavin. C.O.C.

**Photometric Determination of Whiting Capacity of White Pigments**

B. L. Rubinshtain, S. V. Yakubovich, G. S. Bozodanova, and Z. A. Bazilevich

*Lakokrasochnye Material i Ikh Primenenie*,  
(3) (1960) 51–55

*Chem. Abs.*, 55 (23 Jan 1961) 2133

A method based on measurement of the coefficient of colour intensity of a mixture of dry white pigment with Ultramine (C.I. Pigment Blue 29) and determination of the whiting capacity from a calibration curve based on the white pigment. Original colour intensity of the white pigment does not influence the whiting capacity of the pigment. C.O.C.

**Rapid Determination of Lithopones (C.I. Pigment White 5). I—Complexometric Determinations of Zinc and Barium in Mixtures**

M. R. Verma and V. M. Bhuchar *Chem. Z.*, (1958) 11670

*Paint Manuf.*, 27 (1957) 384–385, 389

*Chem. Abs.*, 55 (9 Jan 1961) 1023

Procedures are given for determining Zn and Ba compounds alone or in mixtures. Zn is titrated at pH 6.7 (maleic acid-maleate buffer) with the di-Na salt of ethylenediamine tetracetic acid to a red-to-blue end point (Eriochrome Black T as indicator). Ba is determined at pH 10 ( $\text{NH}_4\text{OH}-\text{NH}_4\text{Cl}$  buffer) by adding excess of the above reagent and back-titration with  $\text{MgCl}_2$ . C.O.C.

**New Methods of Measuring Moisture**

K. G. Gröniger *SVF Fachorgan*, 15 (Nov 1960) 745–760

After discussing the importance of controlling relative humidity during various stages of converting fibres into yarns, and then into fabrics, the author describes moisture absorption isotherms of a number of textile fibres. Since humidity and temp. are so closely interdependent, the author believes that the same instrument should be capable of measuring moisture content and temp. of the material. The apparatus should be simple, robust, reliable, and sufficiently sensitive to detect local variations in moisture content. Several instruments which satisfy some or all these conditions are described and illustrated. L.A.T.

**Identification of Wool Damage by Polarographic Determination of Cystine, Cysteine, and Sulphur Dioxide**

L. Beníšek

*Faserforsch. und Textiltech.*, 12 (Jan 1961) 23-27

Polarographic analysis of wool hydrolysates was carried out using the cathodic cystine and anodic cysteine waves. The presence of sulphur dioxide was shown and its origin is discussed. The cystine, cysteine, and sulphur dioxide contents of wool damaged by alkaline, acid, oxidative, and reductive treatments have been determined, and it is suggested that such results may be used to identify different types of damage.

W.R.M.

**Fastness Testing—Committee on Consumer Protection**

J.S.D.C., 77 (Mar 1961) 112-113

**Determination of the Dry-cleaning Fastness of Dyeings and Printings**

*Textil-Rund.*, 16 (Jan 1961) 12

Standard method SNV 95 8 30 of the Swiss Standards Association.

P.B.S.

**Determination of Sublimation Fastness in Storage of Dyeings and Printings**

*Textil-Rund.*, 16 (Jan 1961) 13

Standard method SNV 95 8 35 of the Swiss Standards Association.

P.B.S.

**Influence of Glossy Surfaces on Reflection Measurements**

A. Brookes

*Die Farbe*, 9 (Sept 1960) 53-62

The measured reflectance of radiation from a surface is dependent upon the geometry of the incident and reflected light and also on the nature of the surface. The influence of gloss on a surface must be allowed for when reflection measurements are evaluated. Consideration is given to the connections between the measured reflectance and the combined effects of specular reflection, and internal scattering and absorption in the sample. Equations are given for the following angular relationships between incidence and measured reflectance—(a) diffuse—0°, (b) 0°—diffuse, and (c) 45°—0°. These equations are given in terms of reflection coefficients at the surface for (1) parallel normal incidence  $r_0$ , (2) diffuse incidence  $r_1$ , and (3) diffuse incidence at the surface from the body of the sample  $r_2$  and, in addition, reflection factors at the air surface  $\rho$ , and internal scattering layers  $R$ , together with the refractive index of the medium  $n$ , and a factor  $\alpha$ , determined by the geometry of the photometer receiver. For example, the reflectance  $\beta$ , for diffuse incidence with measurement at 0° is given by—

$$\beta = \alpha r_0 + \frac{(1-r_0)(1-r_1)1/n^2 \cdot R}{1-r_2 R}$$

A comparison of measured and calculated reflectance values is given for a white vitrolite, a grey enamel, and a pink plastic, both for values of  $r_2$  given by previous workers and for those derived by the author. In the latter case good agreement is obtained between the theoretical and observed values.

E.COATES

**Microbiological Detection of Active Substance in Textiles Proofed Against Micro-organisms**

F. Steinfatt *Melliand Textilber.*, 41 (Dec 1960) 1573-1574

The usual method consists in putting a sample of the material to be tested on a dish with agar nutrient inoculated with the test organism and observing whether a zone free of growth is formed round the sample after some time. It has been found, however, that if a sample has been repeatedly washed this test can give negative results even though the material still possesses antibacterial properties. A new, very sensitive test is proposed in which the material is extracted with boiling water, the wet sample is inoculated with the test organism, and nutrient broth is added after 24 h. In a control test pronounced growth is observed after 2 days, while the treated sample shows no growth or the growth is delayed for several days.

W.M.

Laboratory Dyeing Machine (I p. 218)

Lightfastness of Dyes on Textiles (VIII p. 228)

Control of Moisture Content during Open-width Drying of Fabrics (X p. 231)

**XV—MISCELLANEOUS**

**Colours of Equal Lightness**

G. Wyszecki

*Die Farbe*, 9 (Sept 1960) 3-6

In commenting on the correlation found between computed ratios of  $h/A$  and observed ratios of  $L/A$  (relative brightness  $h = A/A_0$ , the ratio of luminous reflectance of the sample colour  $A$  to that of the optimum colour of the same chroma  $A_0$  and  $L$  = luminous reflectance of gray of equivalent lightness) by Richter (*Die Farbe*, 8 (1959) 101-112), the present author derives the standard deviations (%) between the two sets of Richter's values for  $L/A$  (observed) and  $1/A_0$  (computed), between the values for  $L/A$  and the mean of  $L/A_0$ , and between the values for  $L/A$  (observed) and  $L/A$  computed on the basis of the best fitting conoid. It is concluded that there is little correlation between  $L/A$  (observed) and  $1/A_0$  and hence that  $h$  does not correlate well with lightness. Nor does  $L/A$  (observed) correlate well with  $A$ , but it correlates well with  $L/A$  computed from the preferred conoid.

E.C.

**Metameric Colours with Smooth Continuous Reflection Curves under Sources with Discontinuous Spectral Energy Distribution**

I. Hennicke and W. Münch

*Die Farbe*, 9 (Sept 1960) 7-16

The calculation of spectral energy distributions to match a given colour, in particular the calculation of reflectance curves to match a surface under illuminants A, B, and C, have been considered by Brewer and Holly, and by Wyszecki. Methods have been described, transformation equations detailed and data given to obtain such reflection spectra from tristimulus values. Reference is made to these works and to the difficulties encountered when a discontinuous source such as a Xenon arc is used as a surface illuminant. The earlier methods of calculation give reflection data with distinct undulations corresponding to the line emissions from the source. The calculation involves the estimation of three stimuli functions ( $f_1$ ,  $f_2$ ,  $f_3$ ), which will allow an additive match of the sample stimulus to be made. For these three stimuli there is an infinite choice, and equations are given to allow an immediate decision on a reasonable set to be made. The reflectance ( $R_\lambda$ ) at any wavelength is linearly related to these three functions;  $S_\lambda R_\lambda = a_1 f_1(\lambda) + a_2 f_2(\lambda) + a_3 f_3(\lambda)$ , where  $S_\lambda$  is the spectral energy distribution of the source. The constants ( $a$ ) are calculated from the sample tristimulus values, for which purpose equations are given. The present paper concerns itself with these details and also the modification of the function ( $f$ ) by the source distribution ( $S_\lambda$ ). A function ( $F$ ) is introduced, where  $F(\lambda) = S_\lambda f(\lambda)$ . Examples are given of the calculations for a yellow and a blue colour in comparison with the earlier methods.

E.COATES

**Colour-memory Tests—II**

E. Hellwig

*Die Farbe*, 9 (Sept 1960) 73-111

In part I of this series (*Ibid.*, 7, No. 316 (1958) 65-91) the certainty of recognition of hue at constant saturation was studied by colour-memory tests. In part II consideration is given to saturation at constant hue. A review of the relevant literature is given, which deals largely with the psychological basis for the work. The experimental procedure follows the methods described in part I but uses painted colour samples of five different hues (1G yellow, 7R red, 11P purple, 16B blue, and 22Gr green) at three different degrees of saturation (2, 4, and 6). The hues and saturation spacing were based on Richter's colour system for samples of 46.2% luminance factor. Saturation intervals in units from 0 to 8 were subdivided into five parts, and any one observer gave an estimate to this tolerance of saturation by memory from a given sample. The tests were carried out by 26 observers (13 men and 13 women), 8 of whom had some experience of colour by the nature of their work. The results are presented in the form of nomograms and some statistical interpretation is given. The certainty of recognition of saturation is very variable (with a non-Gaussian distribution), improving with repetition, and to a greater degree with female observers. Remarkable certainty is shown by some observers, but there appears to be no connection here with experience.

E.COATES

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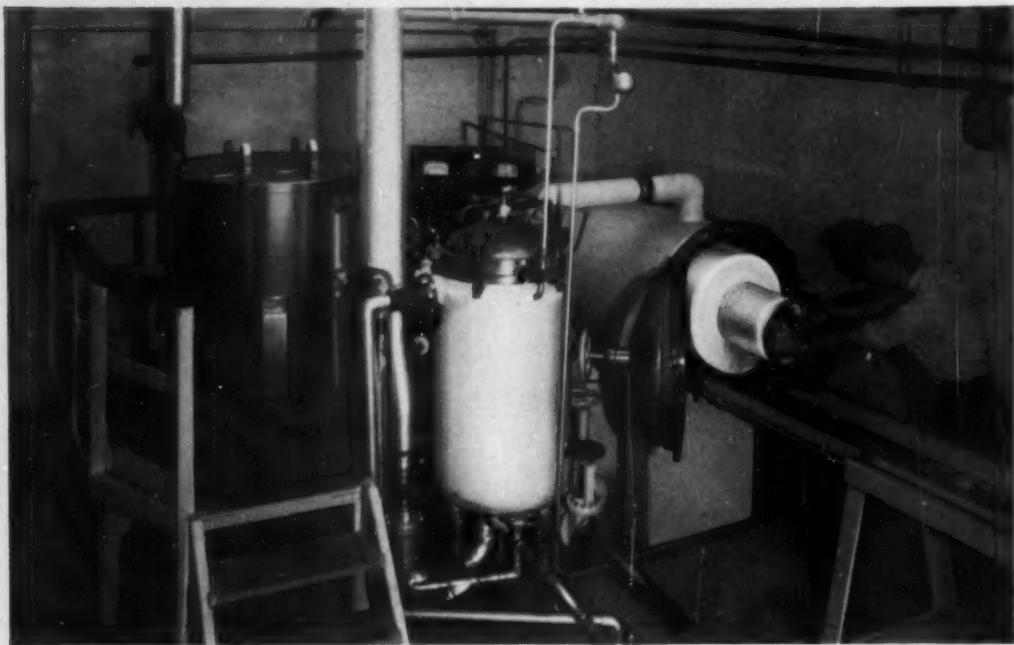
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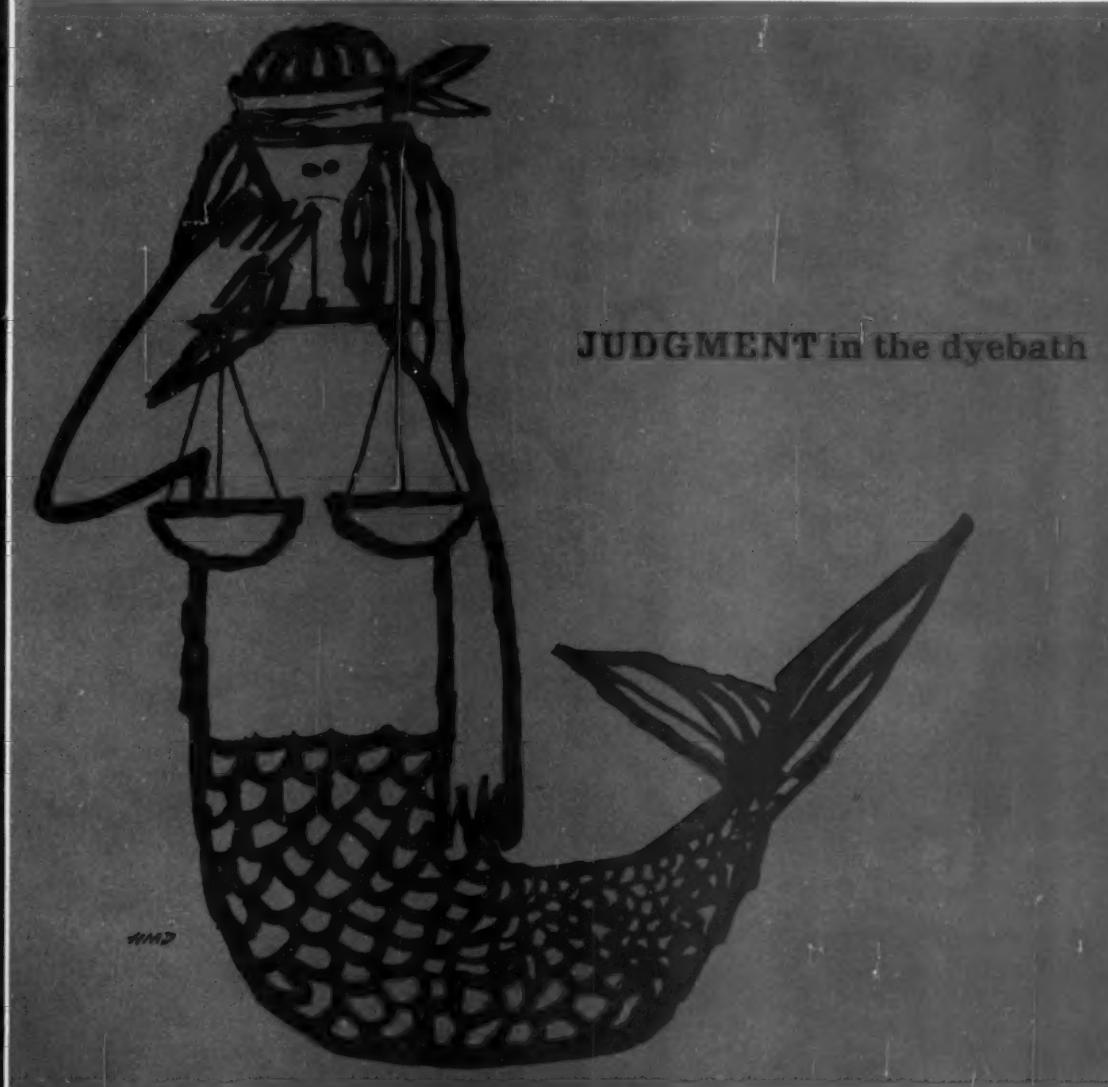
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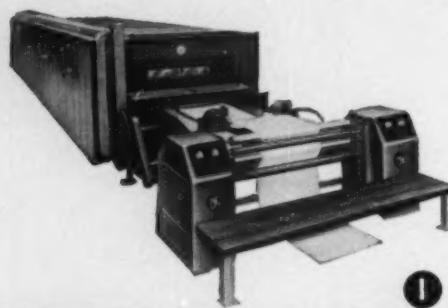
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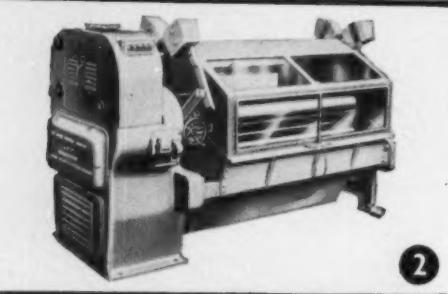
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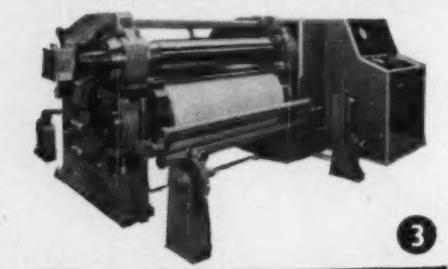
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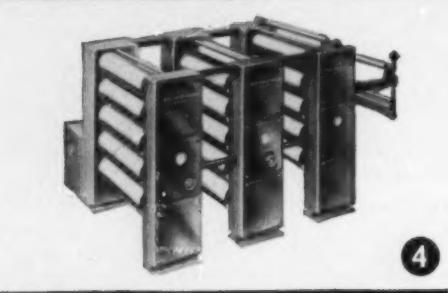
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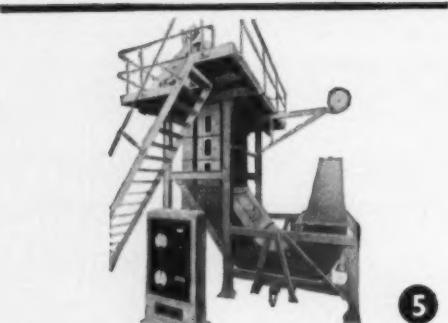
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